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RESEARCH ARTICLE

Effect of contact forms on the wear of hard silicon surfaces by soft polymers

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Abstract: The mechanism of hard surfaces worn by soft polymers is not clearly understood. In this paper, a new hypothesis has been proposed, it holds that the stress acting on the hard surface under certain working conditions is the main reason for wear of the hard surface by a soft polymer. The hypothesis was investigated by changing the contact form between tribo-pairs. For this, friction tests between six polymer spheres and smooth, rough, and inclined monocrystalline silicon surfaces were carried out. The results show that for the same tribo-pair, the silicon surface will not be worn in some contact forms, but in other contact forms it will be worn. We believe the wear of hard surface by a soft polymer is the result of the combined stress state action on the hard surface.

Keywords: polymers; silicon surface; wear; combined stress

1 Introduction

In recent years, polymer has been widely used in engineering because of its light-weight, high specific strength, corrosion resistance, and low friction coefficient [1, 2]. Among these applications, tribo-pairs of hard and soft polymer surfaces are widely used in many fields, such as seals, gears, lathes (cutting), and artificial joints [3–6]. When soft polymer and hard material are used as friction pair, it is usually expected that the soft material is the one that will be worn out, and the theory of surface wear related to material hardness is put forward [7]. The theory is used to predict the wear behavior of materials and, based on this theory, ways to enhance the wear resistance of polymer surfaces were also proposed [8, 9], however, the foundation of these efforts is that hard surfaces are not worn out by the soft polymers.

Since the 1960s, researchers have observed the unusual phenomenon that a metal surface with a very high hardness can be worn out by soft polymers. Specially, a systematic review of these research was presented by Zhang and He in 2004 [10]. Since the wear of hard surfaces by soft polymers becomes an established physical phenomenon, a series of theories have been put forward to explain this unusual phenomenon. Among these theories, there are four primary ones:

1) The mechanism of adhesive wear caused by a polymer–metal reaction was proposed by Vinogradov et al. [11], and developed by Zhang et al. and Wilches et al. [12, 13]. It is thought that when a polymer with active functional groups rubs against the metal surface, the oxidation or heating effects cause the polymer to degrade into highly reactive low-molecular weight compounds that react with the metal surface. This results in a reduction of the wear resistance of the reaction layer and the metal surface will be more

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easily destroyed.

2) Fatigue wear of the micro-asperity peaks on the metal surface was proposed in Refs. [14, 15]. This theory holds that the fatigue wear of micro-asperity peaks occurs during the friction process between a rough metal and the polymer. This results in the separation of hard particles from the metal surface and these particles will be embedded in the soft polymers. Abrasive wear subsequently occurs and results in extensive damage to the metal surface.

3) Hydrogen diffusion assisted wear theory was proposed by Li et al. [16]. The theory holds that friction can promote the dissociation and diffusion of hydrogen in friction pairs containing hydrogen elements. This leads to metal embrittlement, resulting in a significant decrease in its toughness and ductility, which leads to metal wear.

4) The theory of material transfer assisted wear caused by triboelectricity was proposed by Evdokimov et al. [17]. Plastics can be divided into positive and negatively charged materials depending on the charge obtained by friction. It is believed that a polymer with a negative charge will cause decarbonization of the metal surface when they are rubbed against each other. This reduces the wear resistance and results in wear of the metal surface.

It can be seen that there are many controversies on this issue, and existed research is mostly focused on polymer and metal pairs, which has great limitations. If the phenomenon of unusual wear can be found on the hard non-metal surface, it will prompt us to reconsider this problem. But at present, there are no reports of non-metallic hard materials being worn by soft polymers. This paper will report new experimental phenomena on this issue, and these phenomena cannot be explained by the above four theories, so that we can think about this problem more deeply. Monocrystalline silicon has a high hardness and is widely used in the field of micro-electromechanical technology. Its surface can be easily polished, which makes it very suitable for use in investigating surface wear properties.

In recent years, friction testing machines and characterization equipment have made considerable progress, which can bring new research methods for this problem. In this study, the respective wear of polystyrene (PS), polyoxymethylene (POM), polyamide 66 (PA 66), polypropylene (PP), high density polyethylene (HDPE), and pure polytetrafluoroethylene (PTFE) with monocrystalline silicon was tested using various loading motion modes. These six kinds of polymers are often used in practice [18–23], so it is of great practical significance to carry out relevant research and provide guidance for material selection. The surface morphology after wear was carefully measured by scanning electron microscopy (SEM). On the basis of a detailed analysis of the experimental results, a new internal mechanism of soft polymer wear of hard surfaces is proposed.

2 Experimental procedure

2.1 Materials

The polymer materials used in the experiment were PS, POM, PA 66, PP, HDPE, and pure PTFE spheres with a diameter of 12.7 mm, as shown in Fig. 1. The hardness of these balls were measured by a Shore D hardness tester. Before measurement, the balls were milled into a plane and six points were selected to measure, and the results are shown in Fig. 2. The physical properties of six polymers provided by the manufacturer are listed in Table 1. Single-sided

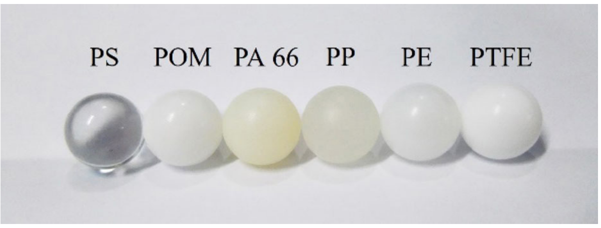


Fig. 1 Appearance of the six kinds of polymer spheres.

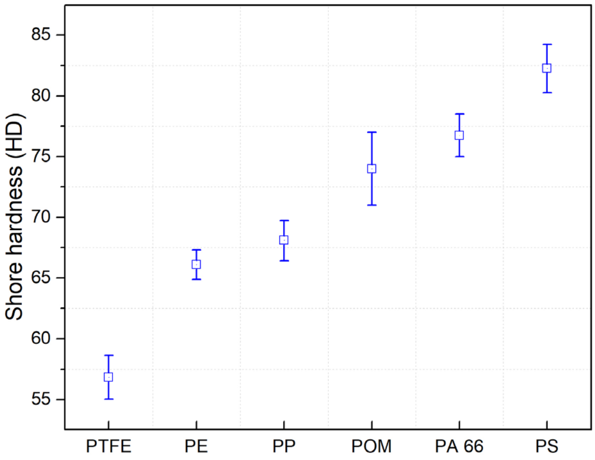


Fig. 2 Shore hardness of the polymer spheres.

Table 1 Physical properties of the six polymers provided by the manufacturer.

Property	Polymer type					
	PS	POM	PA 66	PP	PE	PTFE
Density ($\mathrm{g/cm^3}$)	1.18	1.35	1.12	0.85	0.9	2.17
Elastic modulus (MPa)	3,400	2,600	7,000	900	1,070	150–280
Poisson ratio	0.35	0.39	0.34	0.41	0.42	0.4

polished silicon with high precision was purchased from Tebo Technology Co., Ltd. (Harbin, China). Silicon is treated with HF in industrial production to remove the oxide layer on the surface. As a kind of non-metallic material, monocrystalline silicon has the same hardness as metal, single component (only containing silicon element), no decarburization (previous studies think that decarburization is one of the reasons why hard metal is worn by soft polymer), and it is easier to polish and analyze (which means that we can make a comparison between rough surface and smooth surface experiments). Through the comparison of these factors, monocrystalline silicon is considered to be a very suitable test object in this study. Before the friction test, the surface of the silicon wafer was cleaned with acetone, ethanol, and water to remove contaminants.

2.2 Friction test

The four contact forms used in the friction tests are shown in Fig. 3, these contact forms are actually present in the industry or occur under special working conditions. The reciprocating motion friction tests between the polymer spheres and the smooth and rough silicon surfaces were conducted with a ball-on-plate type on a CETR Universal Micro-Tribometer (UMT-3, USA), as show in Figs. 3(a) and 3(b). A displacement of 5 mm and a reciprocating frequency of 2 Hz were used, and the test duration was 600 s. The normal load applied in this test was 2 N, and each dry friction test was performed under ambient conditions.

Figure 3(c) illustrates the friction test between the polymer sphere and the smooth inclined silicon surface. The surface of the silicon wafer was inclined by adding wedge-shaped blocks with an inclination angle of 20°. The experiment was also carried out using UMT tester. Figure 3(d) shows the rotational motion of the polymer balls on the smooth inclined silicon surface.

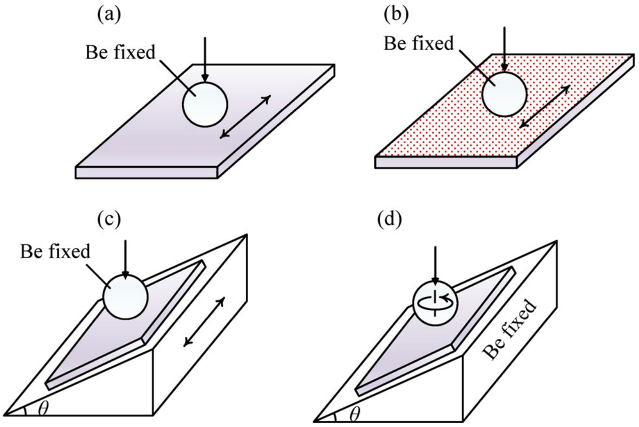


Fig. 3 Schematic of different contact forms: (a) friction between a polymer sphere and a smooth silicon surface; (b) friction between a polymer sphere and a rough silicon surface; (c) friction between a polymer sphere and a smooth inclined silicon surface; and (d) rotation of a polymer sphere on a smooth inclined silicon surface.

The experiment was carried out on a rheometer (Aaton Paar Physica MCR301, Austria) using a three-plate spherical module. The normal load applied in this test was 0.5 N, the rotation speed was set to 50 rpm and the test duration was 10 s, each wear test was repeated at least three times.

What we need to explain here is that this paper explores whether the hard silicon surface will be worn by polymers (because this phenomenon will not occur in common sense), and the reason for this phenomenon. Therefore, we pay more attention to whether the silicon surface will be damaged in various contact forms after a short time of friction test, rather than comparing the wear rate of a hard surface under various contact forms.

2.3 Morphological characterization

Field emission scanning electron microscopy (FE-SEM, Hitachi S-5500, Japan) was used to analyze the morphology and composition of the surface before and after wear. The three-dimensional morphology of the silicon surface and polymer spheres were measured by a white-light interfering profilometer (ZYGONeXView, USA). As shown in Fig. 4, the surface of the flat silicon surface is smooth with a roughness of only 0.001 μm, while the surface of the other substrate is very rough with a lot of micro-asperity peaks and has a roughness of 0.395 μm, the rough and smooth surfaces are two sides of the same silicon wafer. Figure 4(c) presents the two-dimensional profiles of two silicon surfaces for quantitative comparison and subsequent analysis. Similarly, the surface topography of the polymer spheres was also measured, as shown in Fig. 5. For evaluation of the surface roughness of each polymer sphere, the surface was flattened using the software provided with the profilometer.

3 Results and discussion

First, tribological experiments for vertical contact (as shown in Fig. 3(a)) between the different polymer spheres and smooth silicon surface were carried out. The surface morphology of the silicon surface after 10 minutes of friction testing are shown in Fig. 6. It can be seen that the surface of the silicon wafer remains flat and intact after the wear test with PS, PP, and PA 66, and there was no wear on the surface. For surface friction with POM, it can be clearly seen that the wear debris is the transferred polymer from the polymer sphere. In particular, large particles were generated on the silicon surface after friction with PE and PTFE, but the particle composition was not clear. Energy dispersive spectrometry was used to analyze the composition. As shown in Fig. 7, the compositional analysis of the silicon surface rubbed by PE shows that the abraded surface composition was not Si. It can be seen that there is more C in the parts lacking Si, so it can be determined that these particles should be debris transferred from the PE ball. In the same manner, it was easy to prove that the particulate matter in Fig. 6(f) was PTFE because of the lack of Si and the increased presence of the F element in these regions. The silicon surface contained the specific components (C and F) of the polymer spheres used in the friction test, indicating that the surface was covered

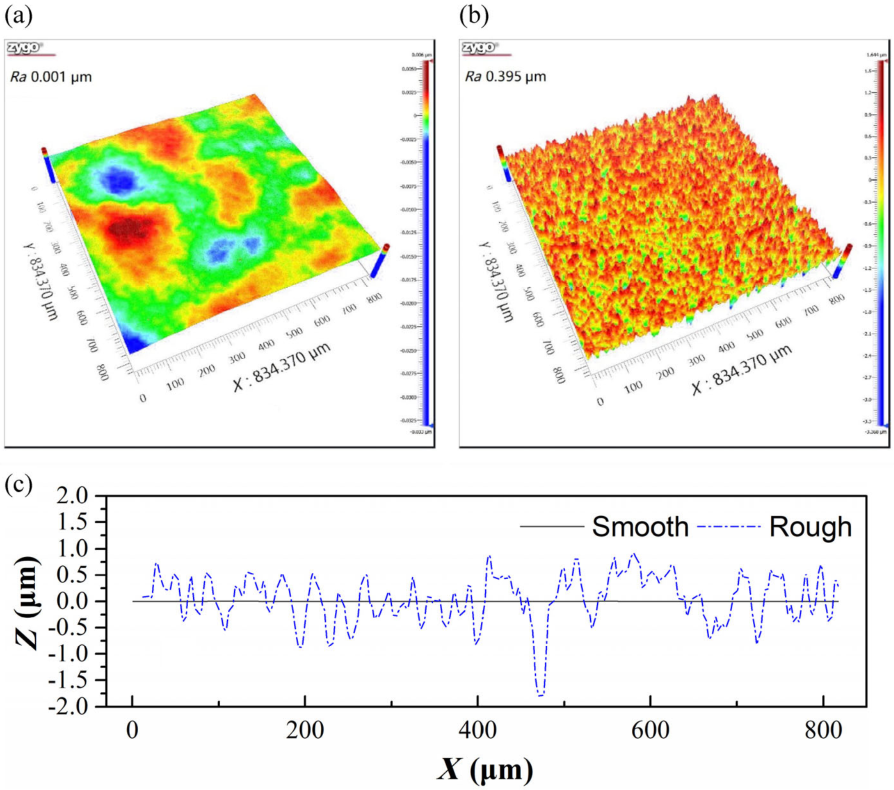
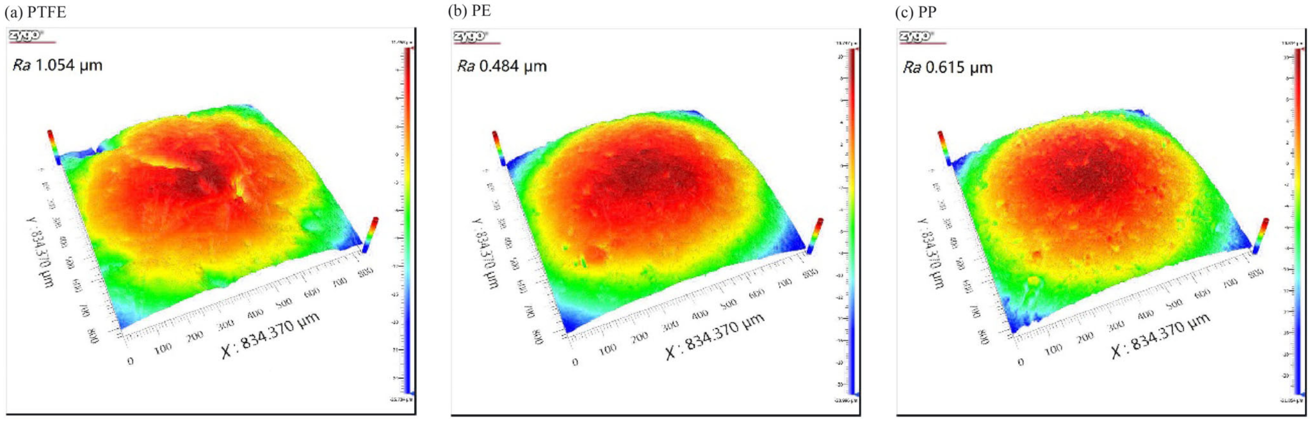


Fig. 4 Three-dimensional morphology of (a) smooth silicon surface and (b) rough silicon surface; (c) surface profile of two silicon surfaces.


(a) PTFE (b) PE (c) PP

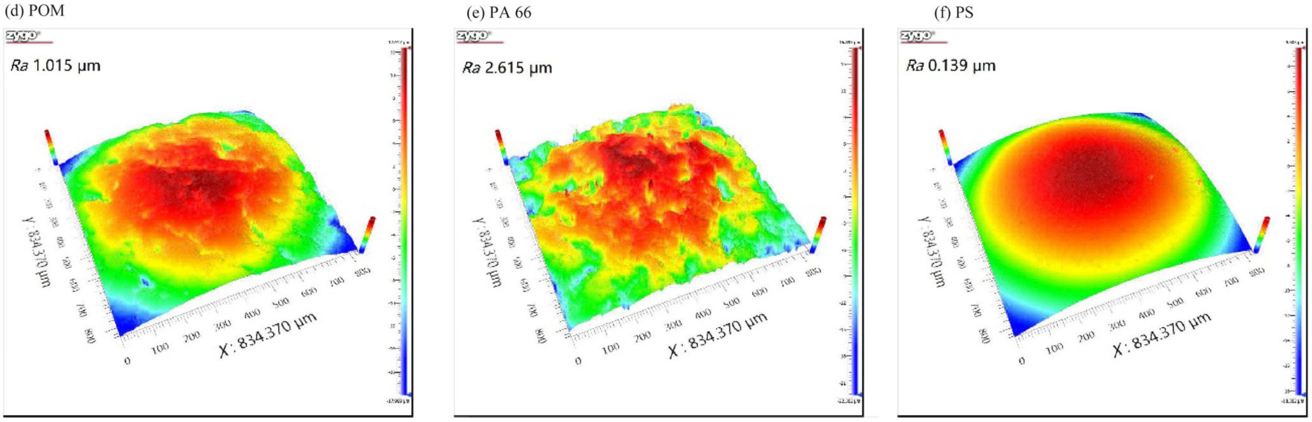

(d) POM (e) PA 66 (f) PS

Fig. 5 Surface morphology of the polymer spheres. (a) PTFE; (b) PE; (c) PP; (d) POM; (e) PA 66; and (f) PS.

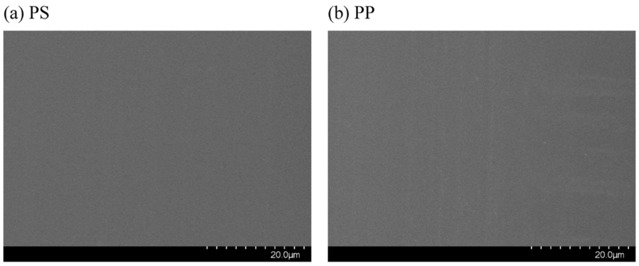
with a thin layer of transferred polymer.

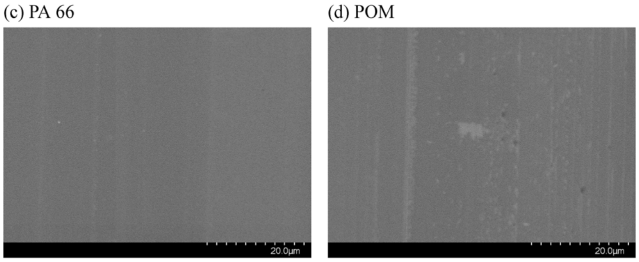
Therefore, the friction test results show that it is appropriate to choose silicon as the research object. During 10 minutes of friction test, the six polymers did not cause any wear on the surface of hard silicon wafers, the softer polymers do not cause the wear of harder surface, which are consistent with common knowledge. However, in the following sections, we will show unexpected experimental phenomena of hard silicon surface wear by the same soft polymer tribopair.

Under the same experimental parameters, the smooth silicon wafer surface was tilted by 20° and similar tribological tests were conducted (Fig. 3 (c)). It was found that when the PP ball was rubbed against the silicon surface, as shown in Fig. 8, serious wear was observed on the surface, the wear appeared as surface cracks and was very clear. It is not difficult to consider that the only difference between two tests was the contact form. Therefore, the different contact form will lead to a great difference in the surface wear behavior, which is very confusing.

To further explore the effect of the contact form on surface wear, we preliminarily tested the rotational friction motion of polymer spheres on a smooth inclined silicon surface (Fig. 3(d)) under the same experimental parameters, serious wear can be observed on the silicon surface. In order to observe the initiation process of surface wear, the load was reduced to 0.5 N and rotated at 50 rpm. After 10 s of the friction test, the surface morphology of the silicon wafer was observed by SEM, as shown in Fig. 9. It can be seen that the silicon surface has slight damage after friction with the PE ball. After the respective friction tests with the PA 66, POM, and PTFE balls, there was only a small amount of polymer adhered to the silicon surface in each case, and no wear was found on the surface. However, there was obvious wear on the surface of the silicon after friction with PS and PP balls, which was covered with cracks and debris.

Here, it can be found that the four theories mentioned in the foreword cannot explain this unusual wear


(a) PS
(b) PP


(c) PA 66
(d) POM

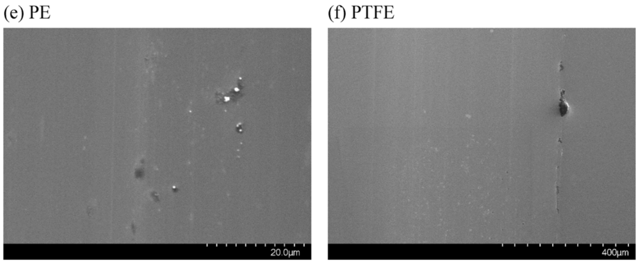

(e) PE
(f) PTFE

Fig. 6 Morphology of the silicon surface after friction with different polymer spheres in the vertical contact mode. (a) PS; (b) PP; (c) PA 66; (d) POM; (e) PE; and (f) PTFE.

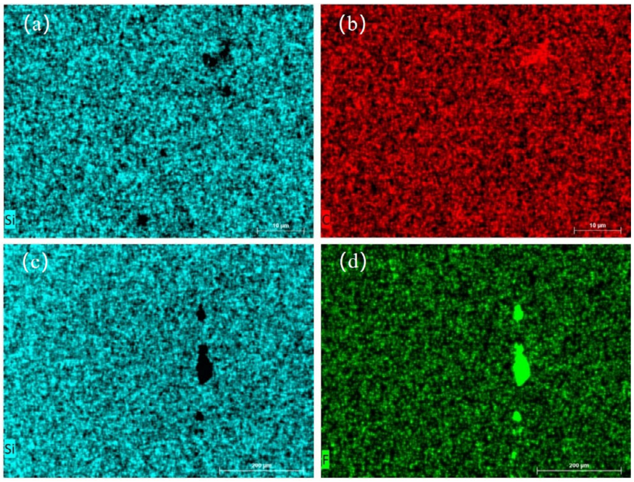


Fig. 7 EDS analysis of the silicon surface. (a) Si and (b) C elements on Fig. 6 (e); (c) Si and (d) F elements on Fig. 6(f).

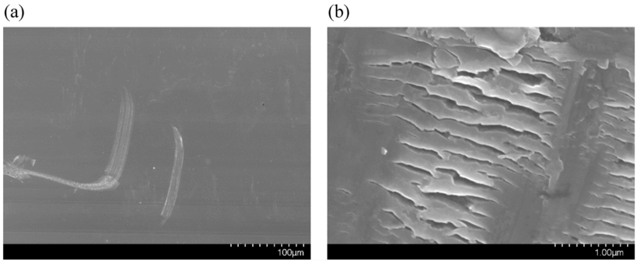
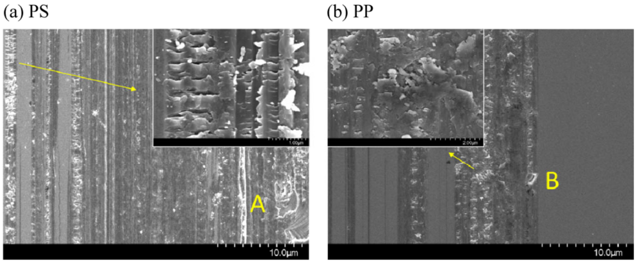
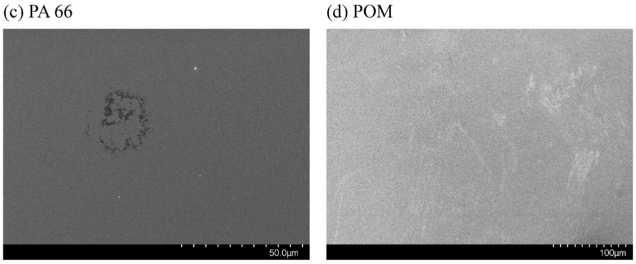


Fig. 8 Wear morphology of the silicon surface after friction with a PP ball with an inclined contact.


(a) PS
(b) PP


(c) PA 66
(d) POM

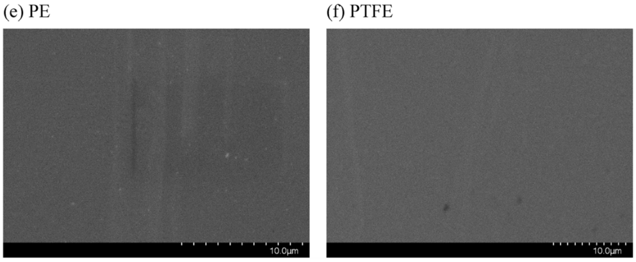

(e) PE
(f) PTFE

Fig. 9 Morphology of the silicon surface after rotational friction with different polymer spheres with an inclined contact. (a) PS; (b) PP; (c) PA 66; (d) POM; (e) PE; and (f) PTFE.

phenomenon. If the above four theories are suitable for this study, the wear behavior of the harder surface should not be controlled by the contact forms. However, in combination with Figs. 6, 8, and 9, it can be found that different contact forms have a great influence on the wear behavior of the harder surface. That is, for the same tribo-pair, the silicon surface will not be worn in some contact forms, but in other contact forms it will be worn. Therefore, there might be other intrinsic mechanisms to control the occurrence of hard material wear caused by soft polymers. We believe that the multi-directional stress acting on the hard surface under certain working conditions is the main reason for wear of the hard surface by a soft polymer. To explain the observed phenomena, the stress state of the surface under several contact forms was analyzed.

For ease of understanding, as shown in Fig. 10(a), when the smooth surface was vertically contacted with the polymer sphere, the surface was only subjected to unidirectional dynamic friction. However, when the surface was inclined, the stress state of the contact area changed significantly and the surface suffered an additional static friction force (Fig. 10(b)). The two friction directions are perpendicular to each other, which is equivalent to the two-way tension force acting on the surface micro-elements of the contact zone (Fig. 10(d)). This can greatly accelerate the initiation and propagation of cracks on the hard surface. When the polymer ball rotates on the inclined surface, the difference with the previous case is that now the point of contact is sliding on the ball and static on the plane. So each surface point on the polymer ball sees intermittent contact, while the silicon is in constant contact. Because of the hysteretic deformation effect of the polymers, the contact stress between the roll-in side of the contact area is greater than that on the roll-out side. Therefore, the hard silicon surface not only bears traction in two directions like previous case, but also suffers a torque in the contact area, which further increases the stress in the contact zone and allows it to be more easily destroyed. Therefore, just 10 s of the rotating friction test under a lower load can still result in considerable wear of the silicon surface (Fig. 9).

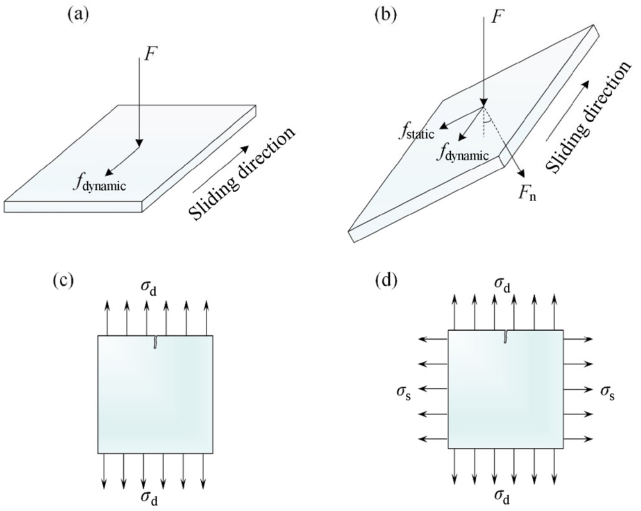


Fig. 10 Schematic diagram of the force decomposition in the friction motion. (a) Friction between polymer sphere and smooth surface with vertical contact; (b) friction between the polymer sphere and an inclined smooth surface; (c) the magnification of the stress state of the element with vertical contact; and (d) the magnification of the stress state of the element with an inclined contact.

In addition, we also observed the occurrence of skidding on the worn surfaces for friction with the PS and PP balls. As shown in Fig. 11, the magnified images of the A and B regions of Fig. 9 show that the sliding direction is at an angle to the friction direction, which may be the direction of the resultant force of the two friction forces on the surface. It can be considered that the wear of the hard surface by a soft polymer is the result of the combined stress on the surface. The combined action of the multi-directional tensile stress aggravates the propagation of surface defects and the initiation of cracks, resulting in the wear of the hard surface. It should be noted that once the hard surface is worn and abrasive particles are produced, the free hard silicon particles can move between the tribo-pairs, as the free third bodies, thus forming the three-body (polymer–silicon particles–silicon) abrasion state. On the other hand, the hard particles can also be embedded in the soft polymer counterpart, thus forming the so-called two body (silicon particles–silicon) abrasion phenomenon [14, 22]. In any case, the subsequent abrasion between the hard particles and the hard surface results in more serious wear of the hard surface.

In this study, friction experiments between rough surfaces and different polymer spheres with vertical contact were also carried out. From Fig. 12, it can be seen that the surface of the silicon can be worn to varying degrees by all the polymer spheres except PTFE. It can be observed that the wear areas are mostly concentrated at the top of the micro-asperity peaks because these areas are stress concentration sites, and the surrounding materials are less restricted; hence, the wear resistance is weaker than that of the matrix material and it is more easily worn out.

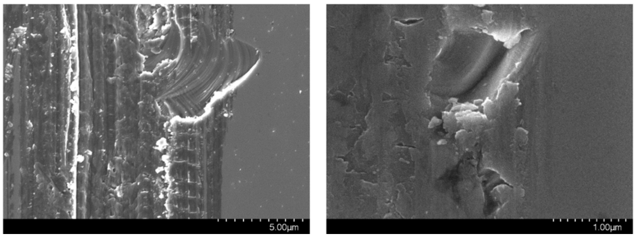

(a) Region A (b) Region B

Fig. 11 Wear morphology of the silicon surface after friction with PS and PP balls.

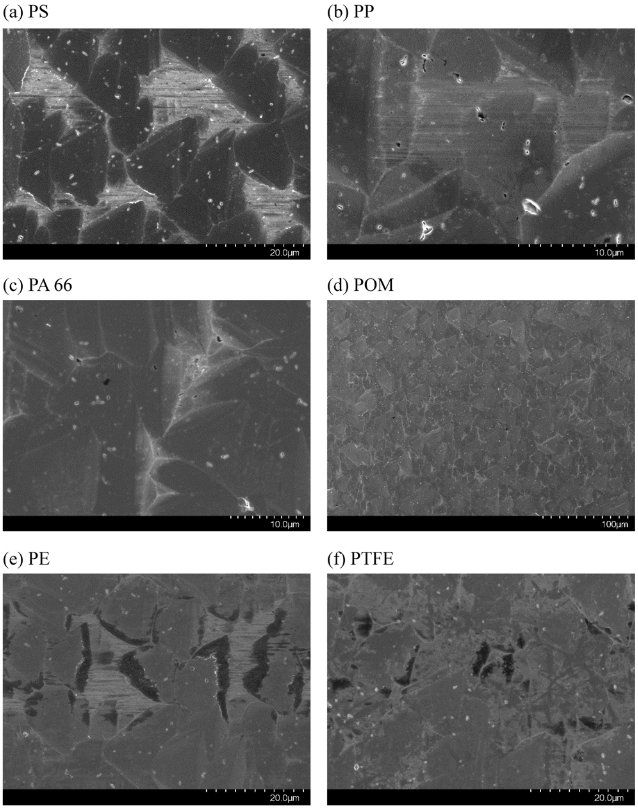

(a) PS
(b) PP
(c) PA 66
(d) POM
(e) PE
(f) PTFE

Fig. 12 Morphology of the rough silicon surface after friction with different polymer spheres with vertical contact. (a) PS; (b) PP; (c) PA 66; (d) POM; (e) PE; and (f) PTFE.

Firstly, as mentioned in many studies, micro-asperity peaks are more susceptible to stress concentrations, which cause wear on hard surfaces due to fatigue [14, 15]. Secondly, what we need to add here is that micro-asperity peaks on the rough surface may bear additional bending moment during friction test, as shown in Fig. 13. When the soft polymer contacts with the hard rough surface, the soft polymer will deform and hinder the movement of the micro-asperity peak [23]. The force of a single micro-asperity peak can be expressed as shown in Fig. 13(b). The normal load and the friction force on the micro-asperity peaks at the contact area can equivalent to an additional bending moment in the root region of the micro-asperity peaks. The effects of the combined stress in the bottom region makes the micro-asperity peak more vulnerable to damage. Therefore, the load on a rough surface is also a contact form in which the surface is subjected to a combined stress. On the other hand, as can be seen from Fig. 13(b), the friction force and its direction on a single micro-asperity peak are different in different positions, the sizes of the micro-asperity peaks are different, the inclination angle changes over the whole contact area, so that the normal load and frictional force (and its direction) change in real time, the combined effect makes the stress of the contact area on the rough surface more directional.

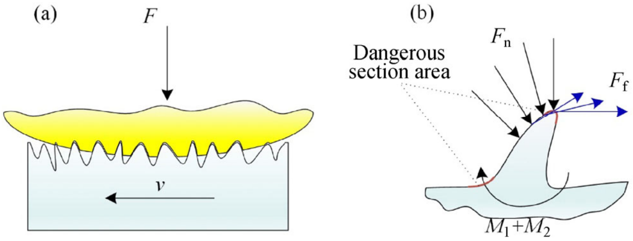


Fig. 13 Schematic diagram of the force decomposition for friction on a rough surface.

In view of the several frictional forms mentioned above, when the surface is subjected to the combined actions of the tensile force, bending moment, and torque, its stress state is more complex, which is more likely to result in wear of the hard surface.

Another important and interesting question relates to the increased wear of the silicon surface by PP, PS, and PE polymer spheres over that caused by the POM, PA 66, and PTFE spheres. Using the data on polymer hardness in Fig. 2, it can be seen that the hardness of the PP and PE polymers are lower than that of POM and PA 66. However, despite the lower hardness, they can cause more serious wear of the hard surface. Obviously, the hardness of the polymer is not the main factor influencing whether the hard surface is worn or not. On the other hand, the composition and structure of the polymers should have an important influence on the wear of the friction pairs. For further analysis, the shape of the debris was investigated in detail for the six polymers. As shown in Fig. 14, it was found that the wear debris of the POM, PA 66, and PTFE polymers are mostly layered, which could form a transfer film on the silicon surface more easily and promote a more uniform stress on the surface. The debris of the PE, PP, and PS polymers were mostly granular, which could more easily result in a concentration of the surface stress and accelerate surface damage. The most typical polymer was PE, and it had spherical wear debris. The spherical particles could change the sliding friction to a rolling friction, which significantly reduces the friction loss on the surface (Fig. 15). However, it may also cause stress concentration at the contact area, which results in surface wear.

To further compare the properties of several polymers, their friction coefficients are plotted in Fig. 15. For the friction coefficients, the effect of the roughness of the silicon surface on the friction coefficients seems to be larger, since the friction coefficient on the rough silicon surface is higher. From the friction coefficients of PP, PE, and PA 66, it can be seen that the magnitude of the one-way friction force on the surface is not a determinant of whether the surface is worn or not. In addition, the friction force of the PP sphere on the smooth surface with a vertical contact is obviously larger; nonetheless, the surface of the silicon wafer is still not worn under these conditions. It is more likely that the surface friction coefficient increases due to the wear of the silicon surface, such as for friction with the PS and PP polymer spheres. It is obvious that the contact mode plays a more important role on surface wear, and the reason has been previously discussed. The pure PTFE polymer appears to be a good selection for avoiding wear on hard surfaces. However, it should be pointed out that pure PTFE has poor wear resistance and commercial products are generally produced using wear-resistant components, which may complicate wear issues. From the perspective of protecting hard surface from wear, POM and PA 66 are better choices as the friction pair for a smooth hard surface according to the results of this study.

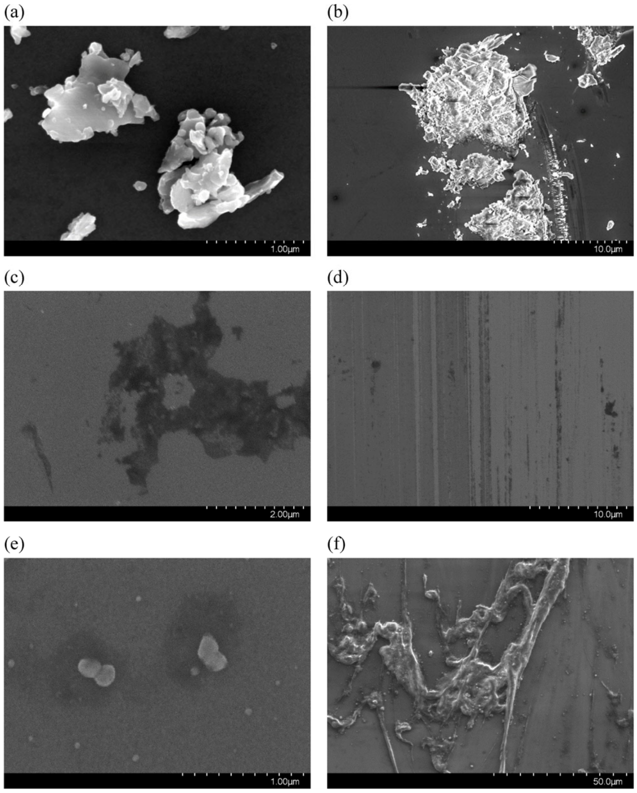


Fig. 14 Morphology of the wear debris of different polymer spheres. (a) PS; (b) PP; (c) PA 66; (d) POM; (e) PE; and (f) PTFE.

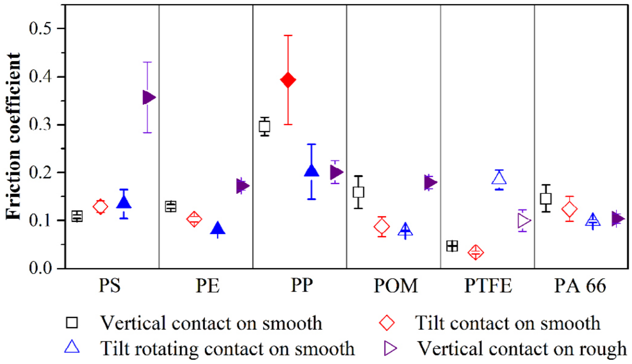


Fig. 15 Friction coefficients of the different polymer spheres with different contact modes (the hollow shape indicates that there is no wear on the silicon surface, while the solid shape indicates that the silicon surface has been worn).

4 Conclusions

In summary, the mechanism of hard silicon surfaces worn by soft polymers was explored in this work. We designed four contact forms to perform friction tests between six soft polymer balls and hard silicon surfaces, it was found that the surface of hard silicon has different wear behavior under different contact forms even for the same tribopair. We put forward a new perspective to explain the observed phenomena. Different contact modes cause hard surfaces to undergo different stress states, which in turn affects the wear behavior of hard surfaces. In addition to dynamic friction, an additional static friction, bending moment or torque will act on the hard surface under certain contact forms. The combined stress acting on the hard surface is the main reason for the wear of hard surface by a soft polymer. Furthermore, the shape of the wear debris also affects the wear behavior of the hard surface. The polymer that produces layered wear debris can more effectively protect the hard surface from wear, but the polymer that produces granular debris could be more likely to cause wear of a hard surface. We believe that the findings of this work will be crucial for studies on the wear behaviors of the hard surface by soft polymer and will also provide useful guidance for engineers to design friction systems in industrial applications.

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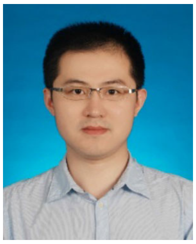

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