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Friction of smooth surfaces with ultrafine particles in the clearance

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Abstract

Friction force between a spherical slider and a smooth flat surface of mica, glass, silicon, sitall, sapphire, metal or diamond-like coating has been measured at reciprocating motion. The slider was made of steel or sapphire. It was revealed that higher surface microhardness of the samples gave lower friction coefficient. An attempt to model friction process of a precision joint in the presence of wear particles in the contact zone was undertaken. For this purpose, ultrafine diamond particles (UFD) were introduced into the friction zone. The friction coefficient increased nonmonotonously with the sample surface microhardness. This fact is explained by abrasive action of the particles and prevailing component deformation in friction. A possibility of reducing the effect of hard particles on friction coefficient has been demonstrated with the surface microrelieves, which contain peak textures. © 2000 Elsevier Science S.A. All rights reserved.

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1. Introduction

Engineering of precise mechanical joints and friction assemblies requires a use of smooth surfaces. In this case the most general requirements for the joints are high contact hardness of the materials, low friction coefficient, sliding stability and long-term operation without lubrication and repairing. The adoption of hard materials to form precise surfaces allows us to meet these requirements. Firstly, such materials can be finished better and, secondly, they experience less deformation under service conditions that results in weaker adhesive interaction. It is commonly agreed that adhesion is the major cause of friction of smooth surfaces [1]. However, there is experimental evidence that the mechanical component of friction (which is plastic deformation of surface layers by the wear particles and plastic deformation of asperities) is dominant in the abovementioned situations [2]. The deformation component of friction significantly increases in the case of debris formation at the interface that gives rise to greater friction coefficient and abrasive wear due to ploughing by wear particles [3]. Furthermore, measurements of friction force

between atomic force microscopy (AFM) tip and a textured surface of magnetic disk show that friction force is governed not only by heterogeneity of the contacting materials but to a larger degree by the surface local tilt at micro- and nanolevel [4].

We have studied the effect of size of dispersed particles and their mechanical properties on friction coefficient and wear rate assuming that abrasive polishing and surface finishing occur [5,6]. It was established that particle mobility is the key factor affecting friction and wear [6]. But, as it was mentioned above, the low friction coefficient and low wear rate in precision joints may be achieved by the elimination of wear particles from the contact region just after their formation. One way to do this is to design surfaces of microgrooves or undulations at the sliding interface for trapping wear particles in the grooves [7]. If there is no way for particles to escape from the contact region they will agglomerate and form larger particles [8-10]. This results in increase of both abrasive wear and deformation component of friction [7].

When abrasive particles sandwiched between two surfaces are loose, the wear rate is less by an order of magnitude than when one material slides against hard protuberances of the counterface. This is because the loose abrasive particle spends most of its time to roll at the sliding interface [5].

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The review of past studies [10] shows that wear particles at the sliding interface dramatically change friction characteristics of the joint. These changes are strongly affected by the way in which the particles are held against the surface and time of interacting with the surfaces.

2. Experimental details

The samples were chosen with fine surface roughness and wide range of hardness. They were made of mica, platinum foil, steel, borosilicate glass, silicon and sapphire. The diamond-like coatings of different thicknesses $(0.1-0.3 \ \mu m)$ were obtained from "PLASMOTECH" research centre, National Academy of Sciences of Belarus.

The formation of wear particles was simulated by spreading ultrafine diamond (UFD) particles over the friction surface. According to atomic force microscopy (AFM) measurements, UFD particles were 5-10 nm in diameter. However, they could agglomerate forming micron-sized particles. In our study we have used suspended UFD particles (0.1 wt.% UFD in alcohol).

The UFD particles were spread over the sample surface by wetting the working area with several drops of the suspension (Fig. 1). Then the base liquid was vaporised for



Fig. 1. 3D (a) and 2D (b) AFM images of ultrafine diamond particles on silicon wafer and a-a cross-section (c) of image (b). Scan size 430×430 nm, height amplitude 17 nm. The arrows on image (a) point at some UFD particles. Values dx and dz on the cross-sections (c) are, correspondingly, relative length and height (in nm) between the profile points marked with the vertical lines.

an hour. Suspended UFD particles are either separated from each other or agglomerated into particles of 40-70 nm in diameter.

The adoption of UFD particles to model wear particles allowed us to attain experimental conditions simplifying the discussion results: (i) the particles are easily measured; (ii) the precise contact is unchanged by them (the maximum peak-to-valley height of the silicon wafer with UFD particles does not exceed 10 nm); (iii) the particles have high hardness and remain undeformed.

The effect of the liquid base of the UFD suspension on the friction coefficient was examined experimentally. Wetting of the surfaces with further drying for an hour resulted in 5% friction coefficient decrease. In addition, friction coefficient was measured for silicon wafer that had vapour-deposited diamond particles. Its value was close to that for suspended UFD. This manifests that UFD particles themselves affect friction but have weak dependence on the way of their spreading.

Friction tests were conducted with reciprocating spherical slider-on-flat tribometer at normal load of 150 mN and sliding velocity 2.2 mm/s. The sliders were of polished bearing steel and sapphire, 4 and 2.2 mm diameter, respectively. The surface roughness of the sliders measured with the AFM was below $R_a = 10$ nm. All experiments were conducted in ambient air at room temperature and relative humidity 30–35%.

Low load, low sliding velocity and small number of cycles enable us to ignore temperature effects, microrelief change due to deformation and wear of surface layers.

The topography of the samples was measured using experimental and computing system "NANOTOP-202" developed at the Metal-Polymer Research Institute of National Aacademy of Sciences of Belarus [11]. The non-contact ac-mode AFM was applied in the system.

The analysis of the tip-sample interaction was performed using the contact AFM "NANOSCAN" (HTE, Russia). A pyramidal synthetic diamond tip was used for probing. The detecting system was based on registration of oscillation characteristics of the piezoceramic cantilever on which the tip was mounted.

3. Results and discussion

3.1. Wear on microscales

The tip of contact AFM was used for the simulation of smooth surface wear by single asperity. Free oscillation frequency of the cantilever was 14 kHz at an amplitude of about 10 nm. The oscillation frequency was maintained at constant level to keep constant load during the scratch tests. To vary the tip pressing force, the frequency set point was shifted. The polished monocrystal silicon wafer was used in the test. From the sample, nanoindentation measurements load was estimated for each scratch.



Fig. 2. One-pass scratching of silicon wafer by the AFM tip. (a) AFM image of the grooves formed under the load of 0.5 mN (groove a), 0.2 mN (groove b) and 0.1 mN (groove c); (A), (B) and (C) pits indented by the AFM tip under the load 0.5, 0.2 and 0.1 mN, respectively (scan size 14×14 micron, height amplitude 115 nm). (b), (c), (d) Cross-sections of the tested area along lines 1, 2 and 3 (a), respectively. Values of dx and dz are in nanometers.

Fig. 2 shows the scratches on silicon wafer resulting from one pass of the tip under a load of 0.5 (a), 0.2 (b) and 0.1 mN (c). Values dx and dz (in nanometers) on the cross-sections are the relative length and height between the profile points marked with the vertical lines. The tip

moved from left to right. The scratch bottom roughness decreased from 12.6 nm for scratch a in Fig. 2b to 4.2 nm for scratch c in Fig. 2c. The mean depth of the scratches decreased and were 105.2, 19.2 and 8.6 nm for grooves a, b and c, respectively. It should be mentioned that static



Fig. 3. Multicycle scratching of silicon wafer by the AFM tip. (a) AFM image of the test area (scan size 14×14 micron, height amplitude 412 nm); a, b, c are reference grooves formed at one-pass scratching under load of 0.5, 0.2 and 0.1 mN, respectively; grooves d, e, f and g formed after 200 cycles of reciprocal tip movement under the load 0.5 mN (grooves d and e) and 0.2 mN (grooves f and g). (b) Cross-section along line 1. (c) Profile of groove d after different number of scratching cycles *n*. (d) Change of groove depth at points A and B (a) vs. number of scratching cycles.

indentation under the same loads at the points A, B and C shown in Fig. 2a resulted in the formation of pits 40, 9.4 and 5.5 nm in depth, respectively. That is, the mean depth is smaller than that of the corresponding scratches. Moreover, depth of the groove scratched under the lowest load increases with the path. This fact can be attributed to the combined stress effect on plastic flow of thin surface layers under normal and shearing stresses. Indentation and wear tests under a load of 0.005 mN did not result in plastic deformation of the sample.

Fig. 3 shows multicycle wear test of silicon wafer. The reference grooves a, b and c were obtained at one-pass scratching under load of 0.5, 0.2 and 0.1 mN, respectively. The grooves d, e, f and g were formed after 200 cycles of reciprocal tip movement in transverse direction. Load was 0.5 mN in case of scratches d and e and 0.2 mN for the grooves f and g. While scratching the surface the profile of the groove bottom was recorded during each pass. The profile cross-sections of the grooves formed with the same loads in Fig. 3b (that is, pairs d–e and f–g) indicate that the difference between the wear test results is less than 20%.

Fig. 3c shows changes of groove d profile after different number of scratching cycles n. One can see different wear of initially flat areas and the pit slopes. Fig. 3d shows the groove depth at point A (the cross point with the groove c) and B depending on the number of cycles. Wear is irregular in time and in some cases it might be negative due to material transfer. Analysing curves in Fig. 3d, it is seen that wear rate tends to stabilise with the number of cycles.

3.2. Friction of smooth surfaces in the presence of UFD particles

For all investigated materials friction coefficient decreased with increasing hardness (Fig. 4). Such behaviour can be explained by both molecular and mechanical theories of friction [1]. In the former case the increase in material hardness results in reduction of comprehensive adhesive parameter of contact due to decrease of the real contact area (RCA) [12]. At the same time the increase of microhardness results in lower contribution of plastic deformation which also decreases the RCA.

From the viewpoint of the mechanical theory, this explanation is not evident. On one hand, the higher the hardness of the softer material the less penetration of the asperities of the harder material into the softer one. On the other hand, higher hardness can cause higher ploughing resistance of the softer material. Depending on the relation between these two factors, friction force can either increase or decrease with the softer material hardness. Furthermore, the friction coefficient is higher for metals in comparison with dielectric materials, i.e., glass and silicon. This may probably result from the higher surface energy of



Fig. 4. Friction coefficient of steel (a) and sapphire (b) sliders against samples of mica (microhardness HB = 0.4 GPa), platinum (HB = 1.3 GPa), glass (HB = 6.0 GPa), silicon (HB = 8.0 GPa) and sapphire (HB = 22.0 GPa).

the former materials. Thus, from the results it is hard to discuss the friction mechanism.

The comparison of the results for steel and sapphire sliders (Fig. 4a and b) shows that the sample deformation has poor effect on friction force. Friction force is inversely proportional to the slider curvature radius if the contact is elastic [13]. However, in our experiments we observed lower friction coefficient when the smaller slider was used. The friction coefficient was greater for the steel slider (Fig. 4a) that had greater curvature radius compared with the sapphire one.

The frictional behaviour changed significantly when the UFD particles were introduced into the contact of interface. It was natural to assume that the particles present in the interface increased the clearance that resulted in weaker adhesion interaction of the surfaces. However, the experimental results showed usually higher friction coefficient at the UFD particle presence. Apparently, hard particles penetrated into the sliding surfaces due to their small size and increased mechanical component of friction. The effect of UFD particles on friction force depends on material hardness of softer flat. Friction coefficient of the sliders against mica (Fig. 4) was slightly affected by UFD that might be attributed to the particles' full penetration into mica. In this case the contact microgeometry remains practically un-



Fig. 5. AFM image of silicon wafer with immovable UFD particles (a) and scanning defects (b, c, d) resulted from motion of the UFD particles after AFM tip. Scan size 800×800 nm, height amplitude 30 nm.

changed and hence friction remains at constant level. The role of scratching increases with hardness of the sample material. The penetration of the particles into the surface may decrease and cannot result in full immersion of the particles into the sample surface. At the same time, it is harder for the particles to abrade the latter. The largest changes in friction coefficient due to the UFD particles at the interface were observed for Si. The effect is slightly smaller for sapphire because of its high hardness which may impede particle penetration under low load.

In those cases, one should take into account penetration of the UFD particles into the slider surface. The penetration depth is less for sapphire slider and it is scratched less as well. Thus, the contribution of individual frictional interaction between the UFD particles and the slider surface to mechanical component of friction is smaller for sapphire slider (Fig. 4b) as compared with that for the steel (Fig. 4a).

It is important to know how firmly the UFD particles are attached to the surface. In our case the interaction with the surface seems to be weak. Some of the AFM images have defects due to particle movement under applied force in non-contact AFM (Fig. 5). Moreover, friction coefficient decreases with the increase in number of cycles. Weakly attached particles are moved off the contact region. This process is strongly affected by the sample surface relief. Table 1 shows results obtained for different number of the slider passes. We can see that even after three cycles of friction with UFD the friction coefficient of the slider against glass or silicon plate restored the value obtained without UFD. Hence, the spherical slider might have moved the particles off the friction zone. When the density of surface microasperities was high (for example, in the case of NiP coating on aluminum, Fig. 6), the friction coefficient increased with the number of passes.

Additional experiment was done for the silicon wafer when the same smooth monocrystal silicon plate changed the spherical slider. Ten-cycle friction with the UFD particles in the interface resulted in formation of scratches of $0.3-0.5 \ \mu$ m width on the wafer (Fig. 7). The grooves at the surface are much greater than UFD particle size. We can suppose that such big scratches were formed initially with agglomerated UFD particles and then with silicon

Table 1 Friction coefficient vs. number of cycles

Sample	Number of cycles	Friction coefficient			
		Steel slider		Sapphire slider	
		clean surface	with UDD	clean surface	with UDD
Silicon	1	0.110	0.340	0.100	0.190
	2	0.110	0.310	0.100	0.170
	3	0.110	0.290	0.100	0.130
Al+NiP	1	0.133	0.133	0.100	0.075
	2				0.107
	3				0.133
Glass	1	0.120	0.210	0.070	0.160
	2		0.170		
	3		0.150		0.147
DLC	1	0.150	0.130	0.130	0.140
	2	0.150	0.128		0.140
	3		0.127		0.150



Fig. 6. AFM image of NiP coating on aluminum substrate. Scan size $12 \times 12 \ \mu$ m, height amplitude 32 nm.

wear debris as well. It is possible that UFD particle agglomerates increased the wear rate. The results agree well with that described in Ref. [2]. We can also see wear debris in the image. The size of the big ones is 300×100 nm. Small ones are about 40 nm in diameter. They can move freely on the surface. Fig. 7a and b shows the motion of the wear particle after the AFM tip.

In practice, the problem is how to reduce friction coefficient to eliminate abrasive wear and prevent dramatic wear of smooth surfaces, particularly silicon. The problem can be solved by coating with a low-shear-strength material or by formation of special texture of the surface. However, high hardness of the surface layer should be retained. For example, friction coefficient between the sapphire slider and the silicon wafers with vacuum PTFE coating of 50-nm thickness was 0.25 while without the coating it was 0.10. PTFE is well known as an antifrictional material but does not reduce friction of silicon surface. It occurs because of significant increase of the contact area due to the higher adhesive parameter of the PTFE and its lower stiffness. These factors have more impact than low shear strength of the polymer.

Another extreme case relates to diamond-like coating (DLC) with high hardness. In our experiments we used



Fig. 7. Successive AFM images of the same area on silicon wafer rubbed against other silicon wafers with UFD particles in the interface. Circled is the silicon wear particle moved by the AFM tip from its initial (a) to final (b) position. Scan size 2.6×3.3 (a) and $2.9 \times 3.2 \mu$ m, height amplitude 97.2 nm (a) and 77.6 nm.



Fig. 8. AFM image of the diamond-like coating. 3D image (a), 2D image (b) and cross-section (c) along line 1 (b). Scan size 5×5 micron, height amplitude 246 nm. Values of d x and d z (c) are in nanometers.

DLC looked like uniform film with quite regular structure of cone-like asperities of 100–150 nm height on the smooth plane (Fig. 8).

On the one hand, mating surfaces separated by big gap should form small real contact area that could result in lower adhesion between the surfaces and lower adhesive component of the friction. On the other hand, average real pressure will be high and hard cone-like asperities will penetrate into the mating surface. This will result in higher deformation component of the friction. The experiment showed (Table 1) that the DLC exhibited higher friction coefficient against the steel slider than the sapphire.

The diamond-like coating was also tested for friction with abrasive UFD particles introduced to the contact zone. Friction coefficient changed insignificantly for both steel and sapphire indenters. However, some fluctuations of the friction coefficient were observed around the initial value obtained without UFD particles. This can be conditioned by secondary and casual factors. Weak abrasive action of the UFD particles when one of the mating surfaces was DLC can be explained by moving them into the gap by the cone-like asperities right at the first pass of the indenter.

4. Conclusions

The study has shown the following.

(1) Sliding process enforces penetration of rigid asperities into softer material that agrees well with the known data.

(2) Rigid particles, for example, wear debris, appearing in the contact zone lead to bigger contribution to the deformation component of the friction. The component is small for soft surfaces (for example, mica), increases with the material hardness, reaches its maximum and then decreases for very hard materials such as sapphire.

(3) It is possible to reduce the abrasive particles' action by formation of discrete relief on the surface, for example, diamond-like coatings with high density of local features.

(4) At friction of smooth surfaces, according to existing friction theories and experimental data, the adhesive component prevails.

(5) Friction coefficient can be reduced with increase in hardness of the mating surfaces.

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