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MODIFICATION OF FRICTIONAL SURFACES OF BEARINGS BY ADDITION OF NANOPARTICLE COMPOSITIONS TO LUBRICANTS

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ABSTRACT *The results of a study of the effect of additives to lubricating oils on changes in the structure of the friction surface and, as a consequence, the tribological characteristics of the "steel to steel" friction pair are presented. The experiments were conducted using a standard "disk-disk" scheme on a friction machine with double cycles of step loading. The material of the disks was bearing steel ШХ-15 with a hardness of 61-63 HRC. Changes in the surface structure were monitored using a binocular microscope. A detailed study of the friction surfaces was carried out using a scanning electron microscope. The microhardness of the friction surfaces was also measured before and after loading. Quantitative changes in the surface relief were determined using a profilometer. The study of the friction surface, conducted using X-ray fluorescence analysis, revealed changes in the composition of the surface layer, which can be explained by the formation of a surface servovite film, which includes elements from the used additive to lubricant (silicon, magnesium, manganese, aluminum, sulfur). The increase in the microhardness of the friction surface when working with the additive from 4.1 GPa to 6.6 GPa, which has a positive effect on the tribological characteristics, is also explained by the formation of a servovite film, which can significantly improve the friction conditions, as well as reduce damage and wear of surfaces. Using scanning electron microscopy, surface waviness oriented at large angles (close to the normal) to the direction of deformation during friction was revealed. It was assumed that the reason for the formation of such a surface relief is microplastic deformation under the action of bias stresses arising in the surface layers of the friction pair. The structural changes observed during friction with the use of a lubricant additive support the assumption of the formation of a ceramic (servovite) film, which has a significantly greater reserve of plasticity compared to a surface with an oxide film, usually present on the surface during friction with a lubricant without an additive.*
Keywords*: grease; additive; surface structure; coefficient of friction; wear; ceramic film; servovite film*

МОДИФІКАЦІЯ ФРИКЦІЙНИХ ПОВЕРХОНЬ ПІДШИПНИКІВ ШЛЯХОМ ДОДАВАННЯ КОМПОЗИЦІЙ НАНОЧАСТИНОК ДО МАСТИЛ

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АНОТАЦІЯ *Надані результати дослідження впливу присадок до мастил на зміну структури поверхні тертя і, як наслідок, трибологічних характеристик пари тертя «сталь - сталь». Експерименти проводилися з використанням стандартної схеми «диск-диск» на машині тертя при двократних циклах ступінчастого навантаження. Матеріалом дисків була підшипникова сталь ШХ-15 твердістю 61-63 HRC. Зміни структури поверхні контролювали за допомогою бінокулярного мікроскопа. Детальне дослідження поверхонь тертя проводилося за допомогою растрового електронного мікроскопа. Також вимірювалася мікротвердість поверхонь тертя до та після навантаження. Кількісні зміни поверхневого рельєфу визначалися за допомогою профілометра. Вивчення поверхні тертя, виконане за допомогою рентгенівського флуоресцентного аналізу, дозволило виявити зміни у складі поверхневого шару, яка може бути пояснена утворенням поверхневої сервовітної плівки, до складу якої входять елементи з добавки до мастила (кремній, магній, марганець, алюміній, сірка). Збільшення мікротвердості поверхні тертя при роботі з добавкою з 4.1 ГПа до 6,6 ГПа, що позитивно позначається на трибологічних характеристиках, також пояснюється утворенням сервовітної плівки, яка дозволяє суттєво покращити умови тертя, а також зменшити пошкодження та зношування поверхонь. За допомогою растрової електронної мікроскопії було виявлено поверхневу хвилястість, орієнтовану під великими кутами (близькими до нормалі) до напрямку деформації при терті. Було припущено, що причиною формування такого поверхневого рельєфу є мікропластична деформація під дією зсуву напруги, що виникає в поверхневих шарах пари тертя. Зміни структури, які виявляються при терті з використанням добавки до мастила, підтверджують припущення про утворення керамічної (сервовітної) плівки, яка має значно більший запас пластичності порівняно з поверхнею з оксидною плівкою, яка зазвичай присутня на поверхні при терті з мастилом без присадки.*
Ключові слова*: мастило; присадка; структура поверхні; коефіцієнт тертя; зношування; керамічна плівка; сервовітна плівка*

Introduction

One of the most important problems of the modern world is the energy problem. The main causes of energy loss and mechanical damage are friction and wear. Friction consumes about 1/3 of the world's produced energy in general and almost 1/2 of the power of vehicles [1]. For example, in a typical passenger car, 79% of the fuel is due to energy loss. [2,3]. About 4/5 of mechanical failures are mainly due to wear and tear of parts of

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mechanisms and machines. In addition, friction also causes serious problems with corrosion of the rubbing surfaces and environmental pollution.

Therefore, reducing friction and wear plays a crucial role in extending the service life of mechanical equipment as well as in saving energy and reducing emissions.

The main and one of the most effective ways to combat friction and wear is lubrication. It is the use of different types and kinds of lubricants that can provide significant energy savings, emission reductions and environmental protection [4].

Sometimes ionic liquids can be used as liquid lubricants [5]. Although such additives demonstrate high tribological characteristics and are environmentally friendly, they have a high cost, which significantly limits their application in industry [6,7].

Additives based on nanoparticles due to their size and surface effects, demonstrate unique physical and chemical properties [8-10]. The use of nanomaterials as lubricant additives can significantly improve the tribological properties of lubricating oil and have a significant impact on the reducing energy consumption and contribute to environmental protection [11,12].

The aim of the work

The aim of the work was to study the influence of nanoparticle additives to lubricating oil on the tribological characteristics of the «steel-to-steel» friction pair and on changes in the structure and relief of friction surfaces. The main interest of the study was the coefficient of friction and wear of the selected friction pair under the influence of nanoparticle additives, which characteristics are described in [13].

Samples and test methods

Tribological tests were carried out according to two experimental schemes.

In the first test, a scheme the standard friction machines were used. The test method was similar to ASTM G77. We used the "disc-disc" test mode (Fig. 1, a), where 1 is the fixed disc and 2 is the moving disc. The speed of the moving disk was 1.3 mps. The diameter of the disks was 50 mm. The disks were made of alloy bearing steel ШХ15, which was heat-treated to hardness 61-63 HRC. This steel is a typical material for bearings, and this type of friction is common for the operation of rolling bearings in the working nodes of various mechanisms. We used two cycles of step loading. During the cycle, the magnitude of loading was increased every 2 minutes of the test. The loading magnitude was changed from 0.2 to 1.0 kN (the step was 0.2 kN). After unloading, the same test was repeated. The graphical recording of the load dependence was performed during the test. Inductive data unit of friction machine was used for friction torque registration. The coefficient of friction (*f*) was calculated using the formula:

$$f = 2M / D \cdot P,$$

where M is the friction torque, D is the diameter of the moving specimen, P is the magnitude of loading.

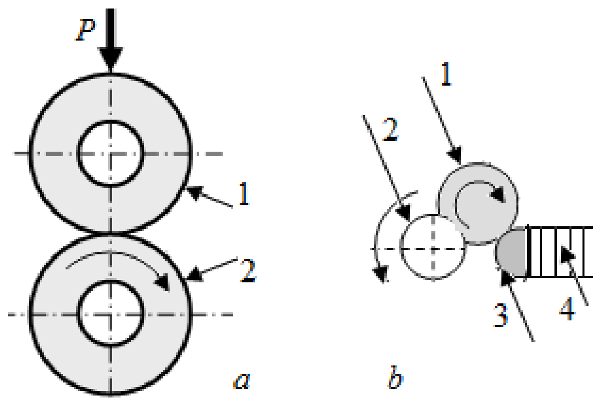

Fig. 1 - (a) disc-disc test scheme; (b) rolling friction bench test

The beginning of fretting was determined as the sharp increase of torque and friction coefficient, and by the appearance of the scores on the working surfaces. The errors in torque and friction coefficient determination ranged from 9% at P = 0.2 kN to 2% at P= 1.0 kN.

In the second experimental scheme, a specially designed test bench was used to perform the rolling friction tests (Fig. 1, b). The load ball (1) was placed on the motor shaft (2) through the rubber coated conductor. As a result, the load ball rolled on the test ball (3) fixed in the holder (4). The wear rate was measured by evaluating the size of the wear marks as a function of the test time. By varying the size and mass of the load ball and the time of the test, the friction conditions can be changed.

Lubrication was performed with MobilGrease XHP222 without additive and with patented additive [13]. Lubrication was performed once just before the start of the experiment.

The examination of the condition of the working surfaces was carried out by means of the binocular microscope under the magnifications x8...50 and by means of the scanning electron microscope (SEM) РЭММА 101-А in the magnification range x30...1000.

Microhardness measurement was made by means Microhardness tester at the loading 50 g.

Measurements of surface roughness were made by means of a roughness tester (type 296) with registration of the average height of roughness.

The surface roughness of rolling elements, raceways and mating surfaces of rolling bearings is typically R_a= 0.63 - 0.32 μm. Therefore, the working surfaces of the disks were machined and reworked according to the 8th class of the State Standard, which corresponds exactly to this roughness. Fig. 2,a shows the initial surface roughness.

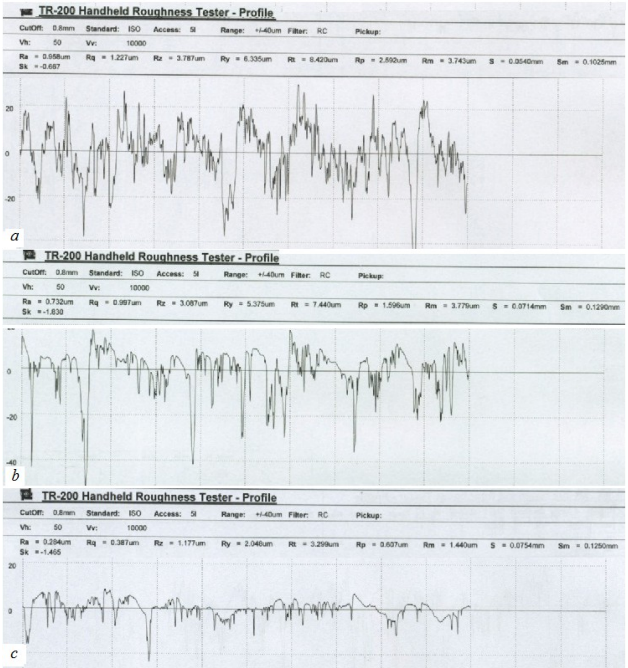

Fig. 2 - Profiles of the initial surface (a) and the surfaces after the friction without (b) and with (c) additive

Experimental Results

Tribological tests results

The dependence of the coefficient of friction on the load during the tests using the grease with and without additive is shown in Fig. 3.

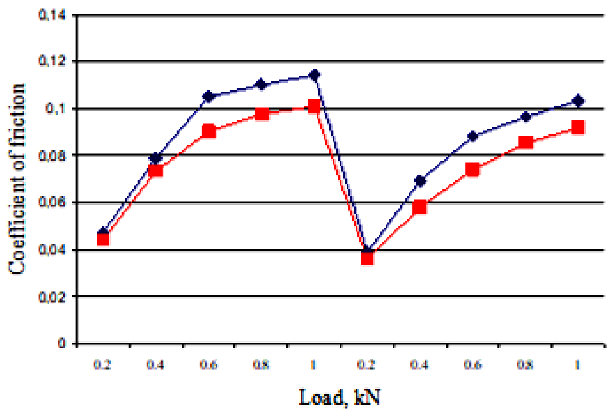

Fig. 3 - Coefficient of friction vs. load (the lower red curve refers to lubrication with additive)

As can be seen from the dependence of the coefficient of friction on the load, the introduction of the additive improved the frictional condition and, as a result, the average magnitude of the decrease in the coefficient of friction is approximately 11%.

The analysis of the wear scar shows that the introduction of the additive into the lubricant reduces the scar size by an average of 10%. This result correlates well with the friction coefficient measurements.

Surface structure study

X-ray fluorescence analysis was performed on the disks tested in grease and in grease with additive. This method allows the determination of the elemental composition of the investigated samples. The depth of the information volume is less than 20 µm for heavy elements and about 10 µm for light elements (e.g. Al). The results of the examination of the initial surfaces after mechanical treatment and the surfaces of the specimens after friction using grease without and with additive are shown in Fig. 4.

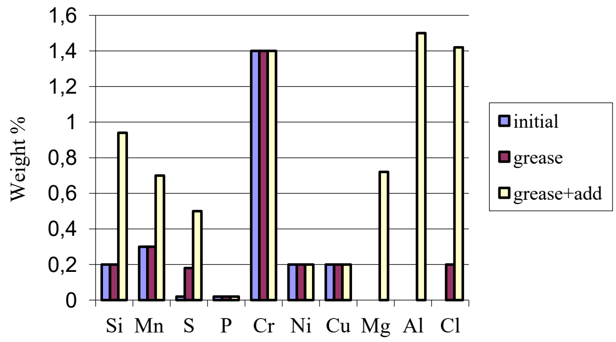

Fig. 4 - Bar graph of surface composition

The observed changes in the composition are related to the influence of the elements present in the grease (e.g., chlorine) and in the lubricant additive (silicon, magnesium, manganese, aluminum, sulfur).

The results of microhardness measurements of the initial surfaces and the surfaces after friction in grease and grease with additive show the increase in microhardness after friction processes. The microhardness of the initial surface was 3.2 GPa, of the surface after friction with grease - 4.1 GPa, with grease with additive - 6.6 GPa. We assume that this fact reflects the contribution of the metal-ceramic film formed by the additive effect to the surface hardening process.

The analysis of the profiles (Fig. 2) of the surfaces in the initial state (a) and after friction in the grease without (b) and with additive (c) shows the essential difference in the surface transformation during the friction process. The initial surface structure represents the rolling and swelling of the mean line. The changes in surface roughness with friction in the grease are related to the cutting off of the sharp raised elements of the surface structure, while the voids remain unchanged. A fundamentally different type of structural transformation of the surface was observed at friction in the grease with the additive. Not only smoothing and flattening of the surface, but also filling of pits and cavities took place during the friction process.

The surface roughness was estimated using the following parameters: R_y – sum of the height of the highest peak and the depth of the deepest groove with respect to the median in the base length limits, R_p – height of the highest peak with respect to the median, R_m – depth of the deepest groove of the profile with respect to the

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median. The results of the average values accumulated from the analysis of 8 - 11 areas of about 5 mm in length of each of the tested specimens are presented in the Table 1.

Table 1 Relief measurements in tested specimens (average values)

Description of the specimen	R_y, **µm**	R_p, **µm**	R_m, **µm**
Initial surface	5.96	2.68	3.28
Friction in grease	4.27	1.37	2.9
Friction in grease with additive	2.58	0.802	1.78

The bar graph (Fig. 5) shows the change in the heights and depths of the elements of the structure of the initial surfaces and surfaces after the friction process using grease and grease with the additive.

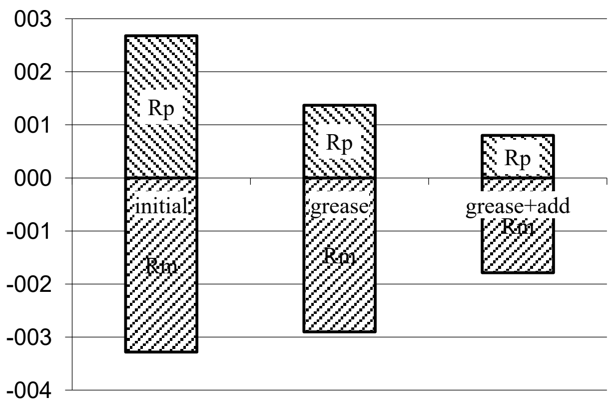

Fig. 5 - Bar graph of the heights (Rp) of the highest peaks and the depths (Rm) of the deepest grooves of the profile for the initial surface and the surfaces after friction using grease without and with additive

The structures of the surfaces of the specimens in the initial state and after the tests using grease lubrication and grease with additive were examined by scanning electron microscopy (SEM). The surfaces of the specimens before the tests showed the relief elements created by mechanical machining, i.e., relief holes oriented along the direction of the machining tool movement (Fig.6, a). Drill holes are arranged with uniform regularity of ~1...5 µm. At the magnifications higher than x100, small cracks and chippings are observed on the surface.

The surface of the specimens subjected to the tests with the grease without additive shows mainly the relief similar to the relief of the surfaces subjected to the mechanical treatment, but it has a less developed and more flattened type (Fig.6, b). The images of these types are typical for the surfaces of steel specimens machined under corrosion wear conditions [4]. (The corrosion wear is the process of fracture of metal surfaces during friction caused by the simultaneous course of plastic deformation process, formation of chemically absorbed films, formation of solid solutions and metal oxide films and their separation from the friction surfaces). It can be assumed that the pictures show very thin oxide films repeating the initial surface relief, which was flattened by mechanical and chemical processes taking place during friction (especially during the running-in period).

The surfaces of the specimens which have been exposed to the tests using the lubricating by the grease with additive demonstrate the relief of the different type (Fig. 6, c). They show the smoother relief, the absence of mechanical processing traces (they differ from the case of corrosion wear during the test using the grease without additive when forming oxide film repeats the relief of initial mechanical processing surface). It testifies that the ceramic (servovite) films forming on the metal friction surface as a result of additive action have the thickness greater than the thickness of oxide film. Their thickness is greater then the height of the roughness of mechanical processing surface. Increased smoothness of the servovite film relief in comparison to the oxide films is one of the factors which stipulate the coefficient of friction decrease and the improvement of other tribological characteristics when the additive is utilized.

Another remarkable feature of the images is the character of the development of defects on the surface of the film, which is connected with the accumulation of pores and their further coalescence, accompanied by the formation of network patterns. Such character of surface defects (crack type) assumes that their development takes place in time, i.e., it is a kinetic process. This is their favorable difference from cracks in oxide layers, which develop at high rates and can be the critical event for the film structure.

The distribution of pores in servovite films accompanied by the formation of mesh patterns may testify to the favorable (uniform) distribution of microstresses in comparison with the formation of the polygonal structure, which is optimal from the point of view of the combination of strength and plastic properties at high-temperature thermo-mechanical treatment of high-strength steels.

We assume that this is the way of realization of the phenomenon of structural adaptability of ceramic (servovite) layers during friction, which causes changes in structure and properties of surface layers in favorable direction at given loading conditions.

Another phenomenon, which is confirmed by significantly higher plasticity in comparison with oxide friction surfaces, is the presence of waviness oriented at large angles (closed to the normal) with the direction of deformation during friction. The source of this waviness pattern formation is microplastic deformation under the influence of displacement stresses occurring in the surface layers of friction pairs. Waviness relief is formed in the zones of crack initiation at the cleavage along the sliding surface under the influence of tangential stresses during the ductile fracture tests of the specimens with sharp stress concentrators. The presence of such microrelief zones is typical for materials with high toughness, and the size of these zones (~10-50 µm) correlates with the magnitude of fracture toughness.

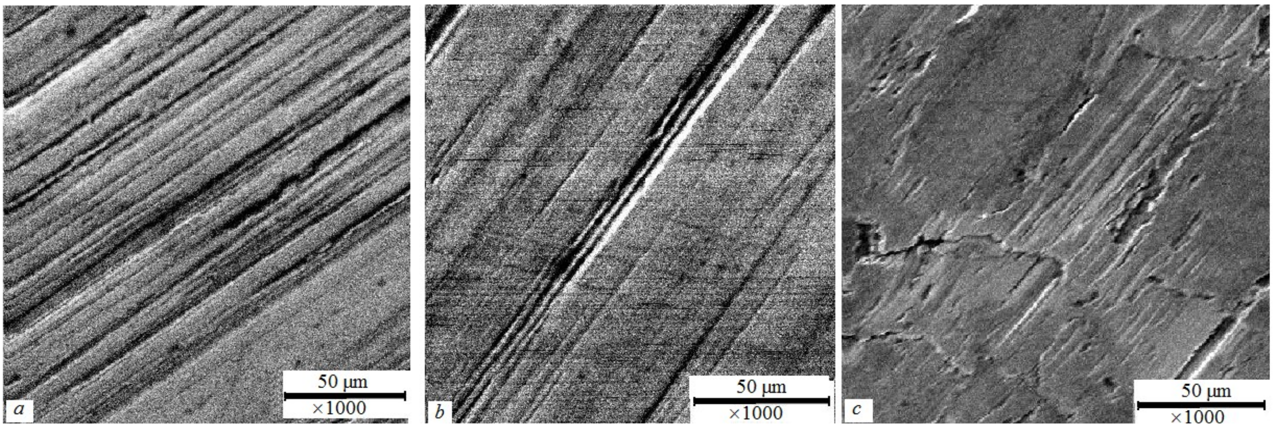

Fig. 6 - Scanning electron microscope images of the initial surface (a) and the surfaces after friction when the grease without (b) and with (c) additive was used

We believe that more pronounced toughness of the film formed during friction in the grease with additive is connected with the presence of magnesium silicate in the additive composition, which plays the role of a catalyst. It slows down the transition of the film substance to a more stable state, promotes the appearance of the intermediate state for which the presence of tough properties is typical. Pores filling and surface flattening are realized due to plastic properties. Depending on the increase of the number of plastic components in the substance, the period of surface layer formation until its complete hardening is increased. During this period, the nanoparticles in the surface layer slide and mix with each other, and the phase transformations stimulated by frictional energy take place.

The above-mentioned reasons prove that the ceramic (servovite) films formed during friction in the grease with additive have a much larger plastic reserve in comparison with the oxidized surface participating in the process of friction with the grease without additive.

Conclusions

It has been shown that the introduction of the additive into the grease during the tribological tests on steel samples leads to the formation of the ceramic-metal film with the thickness < 10 μm on the friction surface. The factors providing the positive influence of ceramic-metal servovite films are: i) modification of the topography of friction surfaces, reduction of their roughness; ii) increase of microhardness, minimization of damageability and improvement of friction condition; iii) minimization of wear processes. The introduction of the additive into the grease has a positive influence on the following operating characteristics of the frictional unit: coefficient of friction, operating temperature, intensity of lubricant degradation, wear, and has a positive effect on environmental safety.

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