

Investigating friction surfaces fabricated with nanodiamonds

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Abstract: The article presents the results of a study of the structure of the friction surface formed when using modified detonation synthesis nanodiamonds with sizes up to 200 and 600 nm in the oil composition. During friction, a layer is formed on the steel friction surface, consisting mainly of wear products of friction bodies and fragments of nanodiamond and oil destruction. The thickness of the layer does not usually exceed 10 μm , but in some cases, an additional layer can completely compensate for wear. The layer is porous and, with simple technology, can always be impregnated with oil, which eliminates dry friction at the start of movement. The diameter of the pores does not exceed 100 nm. The composition of the layer has been studied through atomic force microscopy, X-ray photoelectron spectroscopy, as well as X-ray absorption spectroscopy (XANES) and diffraction using synchrotron radiation, including after etching with Ar^+ ions. The resulting layer, separating the friction bodies, minimizes contact wear occurring at the beginning of movement, during overloads and micro-impacts, and partially or completely restores the geometry of the friction surfaces. Using modified nanodiamonds in the oil composition, it is possible to significantly increase the service life of mechanisms, which is determined by the contact wear of friction pairs.

Keywords: modified nanodiamonds; friction; friction surface structure; wear.

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Исследование поверхности трения, полученной при использовании наноалмазов

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Аннотация: Представлены результаты исследования структуры поверхности трения, образованной при использовании в составе масла модифицированных наноалмазов взрывного синтеза с размерами до 200 и до 600 нм. В процессе трения на стальной поверхности трения формируется слой, состоящий в основном из продуктов

износа тел трения и фрагментов деструкции наноалмазов и масла. Толщина слоя, как правило, не превышает 10 мкм, но в ряде случаев дополнительный слой может полностью компенсировать износ. Слой является пористым и при простом применении может всегда быть пропитан маслом, что исключает сухое трение при начале движения. Диаметр пор не превышает 100 нм. Состав слоя охарактеризован методами атомно-силовой микроскопии, рентгеновской фотоэлектронной спектроскопии, а также рентгеновской спектроскопии поглощения (XANES) и дифракции с использованием синхротронного излучения, в том числе после травления ионами Ag^+ . Полученный слой, разделяя тела трения, минимизирует контактный износ, возникающий в начале движения, при перегрузках и микроударах, частично или полностью восстанавливает геометрию поверхностей трения. Используя модифицированные наноалмазы в составе масла, можно значительно увеличить ресурс работы механизмов, который определяется контактным износом пар трения.

Ключевые слова: модифицированные наноалмазы; трение; структура поверхности трения; износ.

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1. Introduction

Modern mechanisms use lubricants, the main purpose of which is to increase the service life of technical devices. The main factor in the physical aging of components and assemblies is contact wear on friction surfaces. To reduce this, three types of lubricants are used: liquid oils, plastic lubricants, and solid lubricants.

When using oils and plastic lubricants, increased loads can cause hydraulic friction to turn into boundary friction and cause wear on contacting surfaces. In addition, when using oils to start mechanisms after a long period of inactivity, oil starvation occurs, which also leads to accelerated wear on friction surfaces. To reduce wear, anti-friction, anti-corrosion, dispersing, and a number of other additives containing phosphates, sulfates, and chlorides are added to the composition of oils. Their introduction improves the tribological characteristics of oils, but they are environmentally unsafe, since their degradation products are chemically aggressive [1].

Alternative materials that, when added to oils, reduce wear, decrease the friction coefficient, improve the performance characteristics of oils, and are environmentally safe include allotropic forms of carbon, including detonation-synthesized nanodiamonds, fullerenes, and graphene [2–7]. The advantages of such materials have been demonstrated in a number of studies [8–10].

Nanodiamonds have been studied as oil additives for a long time. These materials were first synthesized in 1963 by explosive decomposition of powerful mixtures of explosives with a negative oxygen balance in a non-oxidizing environment. In 1984, under the leadership of Academician G.V. Sakovich, experimental production of detonation nanodiamonds was organized for the first time at the Altai Scientific Production Association [11]. Despite a long history of research, the

mechanism by which nanodiamonds affect the tribological properties of oils and lubricants is still unclear. However, there are some general ideas about the effects of introducing nanodiamonds into oils on the coefficient of friction and surface wear. For instance, the positive effect is thought to be related to an increase in hardness of the friction surface, which is caused by a reduction in grain size due to plastic deformation resulting from the action of nanodiamond particles [12]. It has been assumed that an additional film forms [13] or that the nanoparticles act as “tiny ball bearings” in the contact zone [14]. Thus, nanoparticles in lubricants can transform sliding friction into rolling friction, thereby reducing wear in the contact area. Tribological tests of steel-steel friction pairs (disc-counterbody) with SAE 10W, I-20A and Tr 22 motor oils revealed a reduction in wear of the friction bodies regardless of oil brand when nanodiamonds were present in the oils being tested [15]. At the same time, a porous layer forms on the friction surface.

It has been suggested that nanodiamonds in suspension can form tribofilms on friction surfaces. However, the mechanism by which nanodiamonds act in bearing lubricants and affect their contact durability is complex and requires further research, especially in thin surface layers [16].

This study aims to examine the surface structure and composition of the tribochemical film that forms on a metal surface during friction when modified nanodiamonds (MND) are added to the oil.

2. Materials and Methods

2.1. Starting materials and sample preparation methods

The studies used fractionated MNDs obtained according to the procedure described in patent [16] from RUDD detonation synthesis nanodiamonds with sizes up to 200 and up to 600 nm (Real-Dzerzhinsk

LLC, Russia [17]). MND fractions F1 and F2, according to the dynamic light scattering method, contained particles (aggregates of particles) 4–200 nm and 200–600 nm, respectively [10, 18]. To confirm the sedimentation stability of the particles, samples of base oils with a 10 % MND content were kept for three months before testing. Test samples were obtained by diluting 10 % oil concentrate with the initial I-20A to a concentration of 0.01 % MND. Thus, two types of oils were obtained: modified with nanodiamonds of fraction F1 and fraction F2. I-20A oil (kinematic viscosity 33.7 and 4.5–5.5 mm²·s⁻¹ at 40 and 100 °C, respectively) does not contain additives [19] that could affect the friction surface structure. In order to determine the effect of I-20A base oil on the formation of the friction surface, a series of experiments were conducted, replacing it with glycerin.

In this study, a laboratory “disc-rod” test bench was used. The lower sample was a disc with a diameter of 40 mm and a thickness of 10 mm, made of medium-carbon steel 45. The upper fixed rod (counterbody with a diameter of 10 mm) was also made of steel 45. The friction pair samples were polished to a surface roughness of $Ra = 0.12 \mu\text{m}$. The disc rotated at a constant speed of 250 rpm and was partially immersed in a bath filled with 25 mL of oil.

A thermocouple located in the oil bath was used to record the oil temperature during the test. The load was: 6.5 kg for 10 min, 23 kg for 10 min, 32 kg for 10 min, 40 kg for 30 min, 60 kg for 30 min.

2.2. Methods of analysis

In this work, the friction surface was investigated using atomic force microscopy in a semi-contact mode with a Solver P-47 multimode scanning probe microscope (HT-MDT, Moscow) in air; silicon cantilevers with resonance frequencies of 150–200 kHz were used as probes.

X-ray photoelectron spectra were recorded on a SPECS spectrometer (Germany) equipped with a PHOIBOS 150 MCD9 hemispherical energy analyzer when excited by non-monochromatic Mg K α radiation from an X-ray tube and an analyzer transmission energy of 8 eV (narrow scans). Surface etching with Ar⁺ ions was performed using a PU-IQE 12/28 (SPECS) raster ion gun at an accelerating voltage of 5 kV and an ion current of 30 μA ; the etching rate was 2 nm·min⁻¹. The CasaXPS program was used to process the spectra, and the lines were decomposed using the Gaussian-Lorentzian shape of the maxima after subtracting the nonlinear background using the Shirley method.

X-ray spectra of C K-, O K-, and Fe L-absorption edges were recorded in total electron yield (TEY) XANES mode using equipment from the Russian-German station (channel D161) at the BESSY II synchrotron source (Helmholtz Zentrum Berlin, Germany) (Siberian Center for Synchrotron and Terahertz Radiation [20]). The pressure in the analytical chamber was about 10⁻⁹ mbar, and the size of the X-ray spot on the sample was about 0.2 mm. The Bragg-Bertrand geometry was studied using a weakly diverging X-ray beam and a crystal analyzer, a single-coordinate energy-dispersive counting detector, which made it possible to exclude broadening and peak shifts caused by the complex geometric shape of the sample. The accumulation time per point was 10 seconds. The wavelength used was 1.481 Å, and the scanning step was 0.04 degrees.

3. Results and Discussion

Figure 1 shows the most characteristic results of the three tests of steel friction bodies. Puzyr et al. [15] noted an increase in the mass of the rotating disc during steel-on-steel friction in oil with the nanodiamond content.

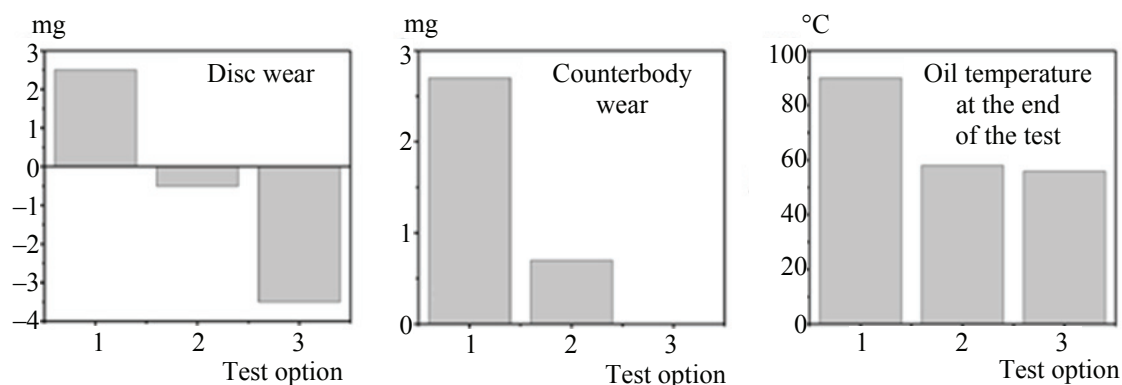


Fig. 1. Results of tests of steel-steel friction pairs (disc-counterbody) depending on the content of the modifying additive. Types of lubricating oils: 1 – base oil I-20A; 2 – oil I-20A with MND fraction F1; 3 – oil I-20A with MND fraction F2. The test duration was 1.5 h, and the spread of wear values for the steel disc and counterbody did not exceed 40 %

The data in Fig. 1 show that in all cases, the addition of MND to I-20A oil leads to a reduction in the physical wear of the friction pair and a decrease in oil temperature. In most cases, disc wear becomes negative, with a more significant increase in disc mass when using larger MND fraction F2. Maximum friction pair wear (disc 2.4 mg, counterbody 2.8 mg) and oil temperature (90 °C) are recorded when using base oil without MND. The use of modified oil with fraction F1 leads to an increase in disc mass (0.1 mg) and a decrease in counterbody wear (0.8 mg). The best result was obtained when using oil modified with fraction F2, when the increase in disc mass was 3.5 mg. For modified oils, at the end of the test a lower temperature was observed as compared to the base oil.

Based on more than 30 experiments, it has been established that the increase in disc mass does not depend on the wear of the counterbody, i.e., it is not caused by the selective transfer of metal from the counterbody to the colder disc. In a number of experiments, a slight increase in the mass of the steel counterbody was observed. In addition, the increase in disc mass exceeds the mass of MND in oil.

The friction surfaces of steel discs were examined after ultrasonic treatment in organic solvents and drying. Fig. 2 shows the topography of the disc surface after testing in oil containing MND fraction F2. Atomic-force microscopy (AFM, see Figs. 2*b* and 2*c*) shows an additional porous layer on the friction track with a thickness of about 10 μm , which is consistent with the calculated thickness from the increase in disc mass, and a pore diameter of about 100 nm (Fig. 2*c*). The porous layer is formed from oxidized wear products of friction bodies, probably due to the abrasive action of MND, accompanied by their destruction, and the thicker this layer is, the higher the content of oxidized iron. The

energy of contact interactions and the growth of the layer depend on the size of the MND clusters. The wear of friction bodies is usually inversely proportional to the thickness of the additional layer.

On the other hand, the spectra show a large, roughly equal amount of aliphatic carbon in the composition of the oil trapped in the pores of different samples. This is also true for glycerin; however, the thickness of the porous layer is smaller and the anti-friction effect of such a lubricant is much weaker. Therefore, the composition of the organic component of the oil affects the formation of the friction surface and the porous layer. Retaining oil in the pores and releasing it under load – for example, during micro-impacts – plays a key role in increasing the effectiveness of the lubricating composition. It should be noted that the porous layer is strongly bonded and cannot be removed without damaging the metal substrate.

As the thickness of the additional oil-enriched layer increases, its growth rate slows down, which is probably due to the passivation of the metal and a decrease in its oxidation rate. This inverse relationship maintains the layer at a certain level, preventing it from disappearing or becoming too large. Its thickness is determined by the size of the MND particles and the energy of the contact interactions. In addition, without any chemical treatment, the products of MND destruction are not chemically aggressive [21], which significantly reduces corrosion wear.

Figures 3 and 4 show the photoelectron spectra of the disc surface outside and on the friction track, respectively. In the first case, even short-term ion etching removes the oxidized layer and exposes metallic iron with an admixture of carbon bound in carbides (line C 1s with a binding energy of 283.2 eV).

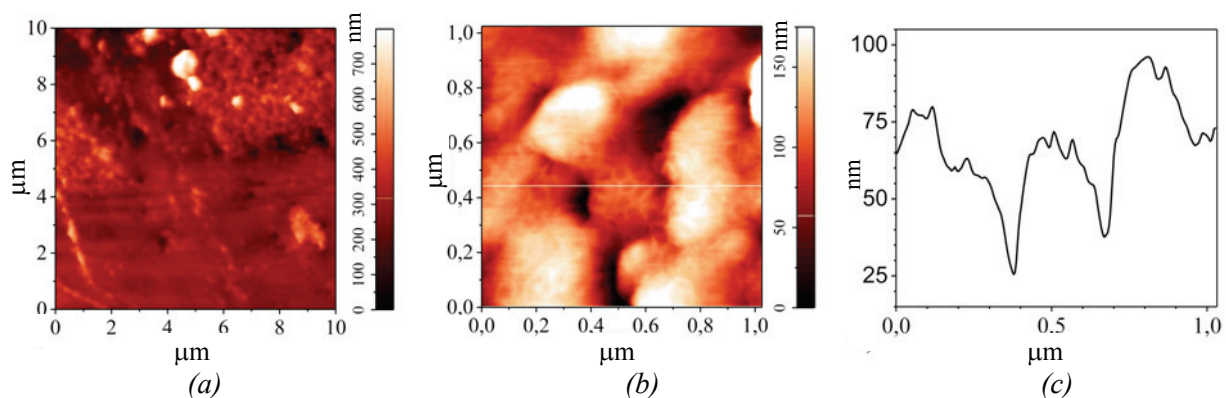


Fig. 2. AFM-images of the rotating disc surface: *a* – friction track boundary; *b* – friction surface relief; *c* – relief profile along the cross section line

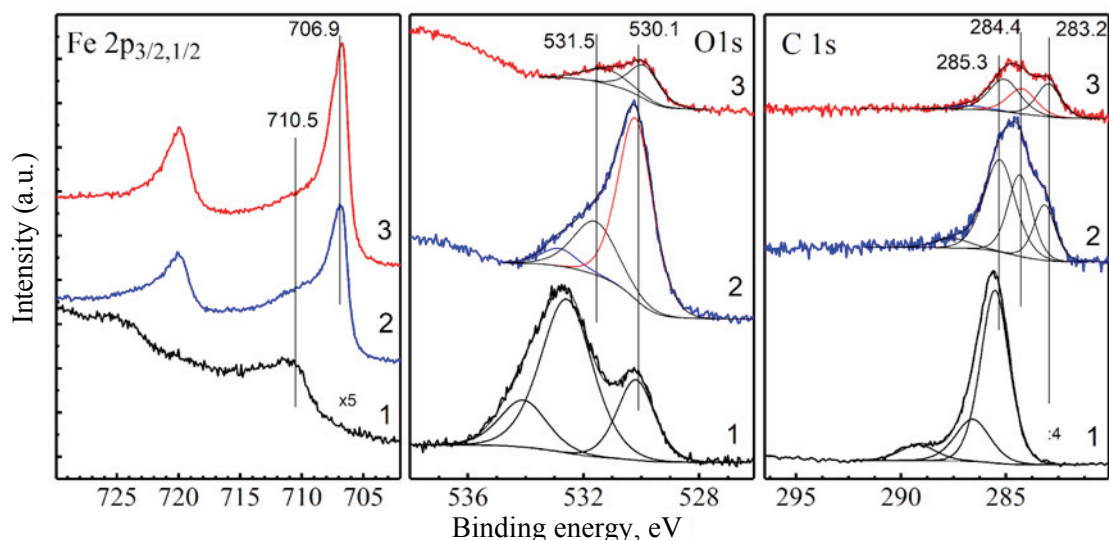


Fig. 3. X-ray photoelectron spectra of Fe 2p, O 1s, and C 1s of the disc surface outside the friction track before (1) and after 2 min (2) and 15 min (3) of Ar⁺ ion etching

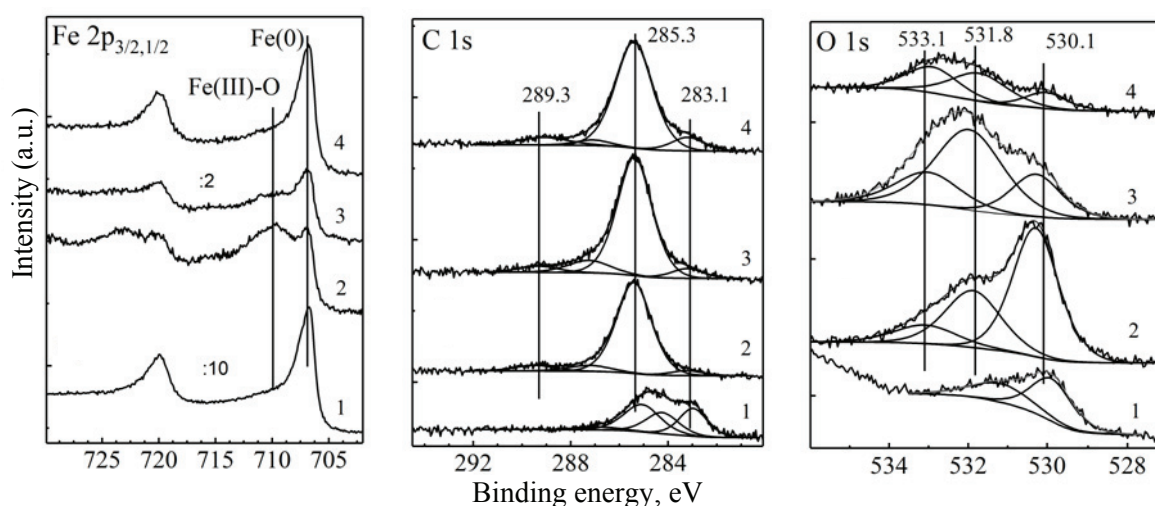


Fig. 4. X-ray photoelectron spectra of iron and carbon after 15 min of Ar⁺ etching outside the friction track – 1 and friction track of the samples: 2 – F1 in I-20A oil; 3 – F2 in I-20A oil; 4 – F2 in glycerin

The spectra of the friction tracks (Fig. 4) before ion cleaning were practically the same, i.e., they were dominated by the lines of carbon and Fe(III) oxihydroxides (about 710 eV), so they are not shown here; instead, the Fe 2p, O 1s, and C 1s spectra after ion etching are considered. The number of O²⁻ and OH⁻ groups (530.0 and 531.5 eV) after etching becomes negligible (Fig. 4c).

The proportion of oxidized iron in the spectra (a broad band with a maximum around 710 eV) decreases after 15 minutes of Ar⁺ etching. Meanwhile, the metal and carbide carbon signals increase for the following samples, in order: outside

the friction band, glycerin fraction F2, modified oil with fraction F2, and modified oil with fraction F1.

Figure 5 shows the Fe L-edge absorption spectra in the soft region in total electron yield (TEY) XANES measurement mode. These spectra characterize thicker layers (10–20 nm) than photoelectron spectra (2–5 nm).

The Fe L₃ maxima consist of lines with energies of 708.2 eV (metallic and carbide iron) and 709.7 eV (iron oxides (+3)). Their relative intensity is consistent with the above conclusions and shows, among other things, that this is not an artifact caused by ion bombardment.

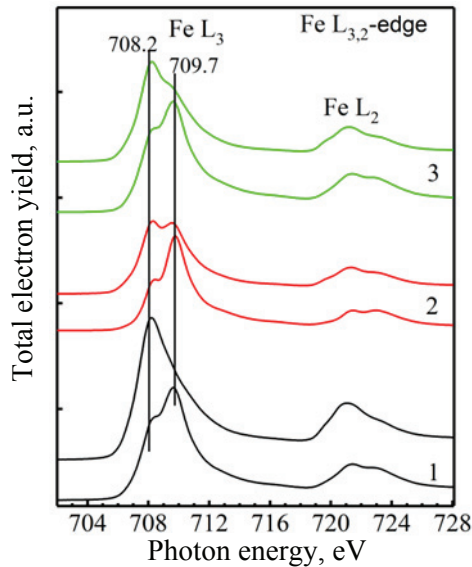


Fig. 5. Fe L_{3,2} absorption edge spectra measured in TEY XANES mode: 1 – outside the friction track; 2, 3 – friction tracks obtained using I-20 A oil with F1(2) and F2 (3) nanodiamond additives. The lower curves were measured before and the upper curves after etching with Ar⁺ ions

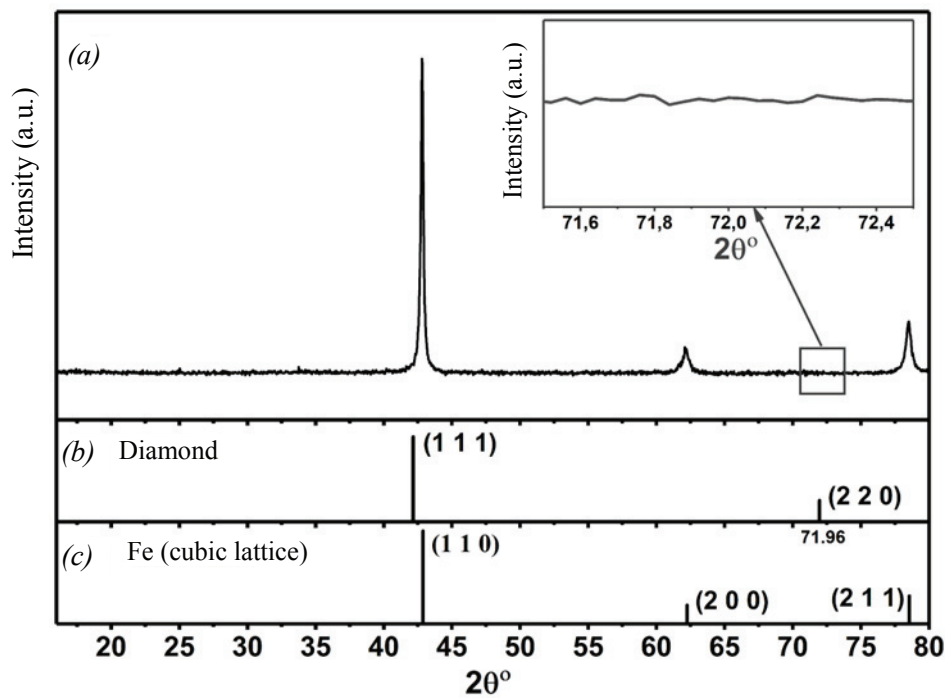


Fig. 6. X-ray diffraction pattern of a friction track obtained in I-20A oil using MND F2 (a). Data from the PDF-2 2014 database for diamond (b) and cubic lattice iron (c) are shown

Figure 6 shows an X-ray diffraction pattern of the surface layer of the friction track obtained using synchrotron radiation (1.481 Å). The X-ray diffraction pattern of the friction surface of the sample obtained using MND only shows reflections that are characteristic of iron with a cubic lattice (Fig. 6a).

4. Conclusion

Two types of oils were obtained as a result of the experimental studies: one modified with nanodiamonds of fraction F1 and the other with fraction F2. A laboratory "disc-rod" test bench was

used for testing with a disc rotation speed of 250 rpm. Using the AFM and XRD methods, it was determined that when detonation-synthesized MNDs with aggregate sizes of less than 600 nm are present in the oil composition, an additional porous layer consisting of iron oxides forms on the surface during friction. This layer retains the oil and MND destruction products. The layer's thickness does not exceed 10 μm. The layer forms due to boundary friction energy and the abrasive action of MND. These factors ultimately determine the presence and thickness of the layer. With the correct selection of the type, size, and concentration of the nanoparticles,

as well as the organic composition, it is possible to achieve virtually wear-free operation of friction pairs, increase the service life of oil, and reduce environmental impact. MNDs can replace environmentally harmful sulfonates in oils and enable friction pairs to operate for longer periods of time.

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7. Conflict of interest

The authors declare no conflict of interest.

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