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Tribocontact surface exploration after friction in hexanoic acid solution*

E. G. Drogan[1], V. E. Burlakova[2]**
[1,2] Don State Technical University, Rostov-on-Don, Russian Federation

Исследование поверхности трибоконтакта после трения в водном растворе капроновой кислоты***

Е. Г. Дроган[1], В. Э. Бурлакова[2]**
[1,2] Донской государственный технический университет, Ростов-на-Дону, Российская Федерация

Introduction. The paper considers the evolution of friction coefficient of the pair of copper - steel alloy under friction in a hexanoic acid solution in various concentrations, and antiwear properties of the steel-steel friction pair in an oil-acidic medium. The work objective is to explore the effect of hexanoic acid additives on the tribological characteristics of friction pairs under the friction interaction in waterborne and paraffin-based formulations.

Materials and Methods. Tribological studies of a brass-steel friction pair were carried out on the AE-5 end-type friction machine. Antiwear characteristics were explored on a four-ball friction machine (FBW) in accordance with the standard GOST 9490–75. When tested at the FBW, the objective parameters of the lubricity of the oiling compositions were: welding load (Pc); wear spot diameter (Dн), critical load (Pк). Roughness parameters of the servovite film were determined through the optical profilometry; its microgeometry and structure at the nanoscale – through the atomic force microscopy.

Research Results. Tribological properties of the brass-steel tribocoupling in aqueous media and steel-steel one in petroleum paraffin-based media are studied. The dependence of the frictional characteristics of the brass-steel friction pair on the concentration of carboxylic acid is established. Its optimum concentration is specified, which provides the effect of wearlessness. A decrease in surface roughness is revealed as a result of the frictional interaction of a brass-steel friction pair in the hexanoic acid solution compared to the initial friction surface due to the formation of a sufficiently dense layer from fine-grained copper clusters with tight particle-size dispersion. The tribological characteristics of a steel-steel friction pair were found to depend on the composition of the lubricant. It is shown that the dependence of the size of the

Введение. Работа посвящена исследованию эволюции коэффициента трения пары сплав медь-сталь при трении в водном растворе капроновой кислоты различной концентрации, а также изучению противоизносных свойств пары трения сталь-сталь при трении в масляно-кислотной среде. Целью данного исследования являлось изучение влияния добавок капроновой кислоты на трибологические характеристики пар трения при фрикционном взаимодействии в составах на водной основе и на основе вазелинового масла.

Материалы и методы. Проведены трибологические исследования пары трения латунь-сталь на машине трения торцевого тира АЕ-5. Исследование противоизносных характеристик проводилось на четырехшариковой машине трения (ЧШМ) в соответствие со стандартом ГОСТ 9490–75. При испытаниях на ЧШМ объективными параметрами смазывающих свойств смазочных композиций являлись: нагрузка сваривания (Рс); диаметр пятна износа (Dн), критическая нагрузка (Рк). Параметры шероховатости сервовитной пленки определялись с помощью оптической профилометрии; ее микрогеометрия и структура на наноуровне — с помощью атомно-силовой микроскопии.

Результаты исследования. Изучены трибологические свойства трибосопряжения латунь-сталь в водных средах и сталь-сталь в средах на основе вазелинового масла. Установлена зависимость фрикционных характеристик пары трения латунь-сталь от концентрации карбоновой кислоты. Обнаружена ее оптимальная концентрация, обеспечивающая реализацию эффекта безызносности. Выявлено уменьшение шероховатости поверхности в результате фрикционного взаимодействия пары трения латунь-сталь в водном растворе капроновой кислоты по сравнению с исходной поверхностью трения вследствие формирования достаточно плотного слоя, образованного мелкозернистыми кластерами меди с малым разбросом частиц по размеру. Обнаружена зависимость триботехнических характеристик пары трения сталь-сталь от состава смазочной среды. Показано, что зависимость размера диаметра пятна износа от содержания кислоты в

* The research is done within the frame of the independent R&D.
** E-mail: ekaterina.drogan@gmail.com, vburlakova@donstu.ru
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wear scar diameter (WSD) on the acid content in the base oil is nonmonotonic in nature with a pronounced minimum at a concentration of 0.1 mass. %. The critical load (Pк) at a content of 0.05 and 0.1 mass. % increases by 32%, welding load (Pc) - by 27%.

Discussion and Conclusions. As a result of the tribological studies of a brass-steel friction pair in the hexanoic acid solution, it has been found that the optimum acid molar concentration in the lubricant composition is 0.1 mol/L. Under the frictional interaction of a brass-steel pair in the hexanoic acid solution, an antifriction copper film is formed on the friction surfaces, which contributes to a sharp decrease in the friction coefficient to 0.007 and metal wear of the friction pair to 25 times. As a result of the frictional interaction of a brass-steel friction pair in the hexanoic acid solution, a decrease in roughness is revealed compared to the initial friction surface. It is found that the frictional interaction of a brass-steel pair in the hexanoic acid solution causes a significant modification of the friction surface as a result of the deposition of finely dispersed copper clusters occurring in the lubricating medium composition and forming a servovite film. As a result of studies, it is found that the dependence of the WSD size on the acid content in the base oil is nonmonotonic in nature with a significant minimum at a concentration of 0.1 mass. %. It is shown that the addition of 0.1 mass. % of hexanoic acid into the lubricant composition exhibits the smallest wear of the steel-steel tribological pair, the WSD decreases to 0.497 mm, the critical load (Pк) and the welding load (Pc) increase by 32% and 27%, respectively.

Keywords: friction coefficient, selective transfer, servovite film, wear scar, welding load, critical load

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базовом масле имеет немонотонный характер с наличием ярко выраженного минимума при концентрации 0,1 масс. %. Критическая нагрузка ($Р_к$) при содержании 0,05 и 0,1 масс. % увеличивается на 32%, нагрузка сваривания ($Р_с$) — на 27 %.

Обсуждение и заключения. В результате трибологических исследований пары трения латунь-сталь в водном растворе капроновой кислоты выявлено, что оптимальной молярной концентрацией кислоты в составе смазки является 0,1 моль/л. При фрикционном взаимодействии пары латунь-сталь в водном растворе капроновой кислоты на поверхностях трения формируется антифрикционная медная пленка, способствующая резкому снижению коэффициента трения до 0,007 и износа металлов пары трения до 25 раз. В результате фрикционного взаимодействия пары трения латунь-сталь в водном растворе капроновой кислоты выявлено уменьшение шероховатости по сравнению с исходной поверхностью трения. Обнаружено, что фрикционное взаимодействие пары латунь-сталь в водном растворе капроновой кислоты приводит к значительной модификации поверхности трения в результате осаждения мелкодисперсных кластеров меди, образующихся в составе смазочной среды и формирующих сервовитную пленку. В результате исследований установлено, что зависимость размера диаметра пятна износа от содержания кислоты в базовом масле имеет немонотонный характер с наличием ярко выраженного минимума при концентрации 0,1 масс. %. Показано, что добавление 0,1 масс. % капроновой кислоты в состав смазочной композиции обнаруживает наименьший износ трибопары сталь-сталь, диаметр пятна износа при этом снижается до 0,497 мм, критическая нагрузка ($Р_к$) и нагрузка сваривания ($Р_с$) увеличиваются на 32% и 27 % соответственно.

Ключевые слова: коэффициент трения, избирательный перенос, сервовитная пленка, пятно износа, нагрузка сваривания, критическая нагрузка.

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Introduction. The issues of friction and wear are the basis of tribology, the science of the interaction of the mating surfaces of contacting bodies in relative motion. In the aerospace and engineering industries, the reduction of friction and wear is one of the priorities [1-3]. Currently, to reduce friction and wear, special attention is given to lubricants with antifriction additives, which are used as metals or metal oxides with particle sizes in the nanoscale [4–7]. It is found that among the metals used as modifiers or metal plaque additives, copper shows a great tendency to reducing friction and wear as a result of the formation of a metal film with low shear strength on rubbing surfaces on the steel surface [4]. Under friction, such a film prevents direct contact of steel surfaces. Studies on the tribo-conjugated surfaces after friction at the nanolevel establish a dependence of the evolution of the friction and wear factor of a tribopair on the morphology and physicomechanical characteristics of the antifriction film [8, 9].

It should be noted that oil-based lubricants with nano-additives demonstrate improved tribological characteristics, but their application inevitably causes the environmental pollution. Their reuse is not possible.

In this regard, this study objective was to study impact of the hexanoic acid additives on the tribological characteristics of friction pairs under the frictional interaction in water-based and petrolatum-based formulations.

Materials and Methods. The evolution of the friction factor of the system “brass 59 - aqueous solution of carboxylic acid - steel 40X” was studied on an AE-5 end friction machine with a rotation speed of 180 rpm under an axial load of 98 N for 10 hours in the laboratory “Hybrid functional materials based on graphene”, REC “Materials”. Hexanoic acid with a concentration of 0.025-0.5 mol/l was used as an organic component of the lubricating composition.

The investigation of anti-wear characteristics was carried out on the FBW on GOST 9490–75 standard. The friction pair on the steel-to-steel FBW was the point contacts of the balls. Balls for tests were made of bearing steel ShKh-15 on GOST 801–78 and thermally processed to a hardness of HRC 62–66. The ball diameter was d=12.7 mm. When tested on the FWM, the objective parameters of the lubricating properties of the lubricant compositions were as follows: welding load ($P_с$); wear scar diameter (WSD) ($D_и$), critical load ($P_к$). The tests on the FWM were carried out under two modes: tests for 3600 seconds at constant load to determine the wear rate of the test samples through measuring the WSD of each of the three balls using the microscope MMU-1 No. 660002; tests for 10 seconds at the increased load before welding the balls to determine the values of $P_к$, $P_с$. The resulting numerical values were approximated by the least square method. As a lubricating composition (emulsion) for testing on the FBW, paraffinic oil with addition of various concentrations of hexanoic acid was used. The tests were carried out at an acid concentration of 0.025–05 mol/l in the base paraffinic oil.

To determine the thickness of the servovite film obtained on the surface of the tribocontact as a result of the frictional interaction of the brass-steel friction pair, as well as its roughness parameters, we used the Contour GT-K1 optical profilometer with the Vision 64 analytical software installed in the REC “Materials” (nano.donstu.ru). The measurements were carried out using vertical scanning interferometry (VSI) with a scanning speed of 0.1 µm/s, with the RMS repeatability of 0.01 nm.

The topography of the servovite film surface was studied through the atomic force microscopy (AFM). The film surface was scanned using an atomic force microscope of the PHYWE Compact brand under the tapping mode with a single-crystal silicon probe with an aluminum coating.

Research Results. Long-term evolutionary tribological studies of a brass-steel friction pair in an aqueous solution of hexanoic acid establish a dependence of the tribological characteristics on the concentration of acid in a lubricating medium. The analysis of the variation of the brass – steel pair friction coefficient in an aqueous hexanoic acid solution with a concentration of 0.025 and 0.05 mol/L specifies rather low values up to 0.07 [8, 9]. A further increase in the acid concentration in the lubricant to 0.2 and 0.5 mol/L, on the contrary, leads to an increase in the friction factor (Fig. 1).

As follows from the results obtained, during the burn-in period of the tribopair, there is a tendency to decrease in the friction factor. However, with the introduction of a high acid concentration, corrosion processes on the contact surface are also initiated. The application of hexanoic acid in a lubricant composition with concentration of 0.1 mol/L provides the lowest values of the friction coefficient up to 0.007 during lengthy tribological tests, the formation of a servovite film and the transition of the system to anti-wear friction [10, 11]. At the same time, wear of a brass-steel friction pair is reduced by up to 25 times, and on a tribo-coupled surface as a result of a selective transfer during friction, a copper film is formed with different roughness and density of surface coating.

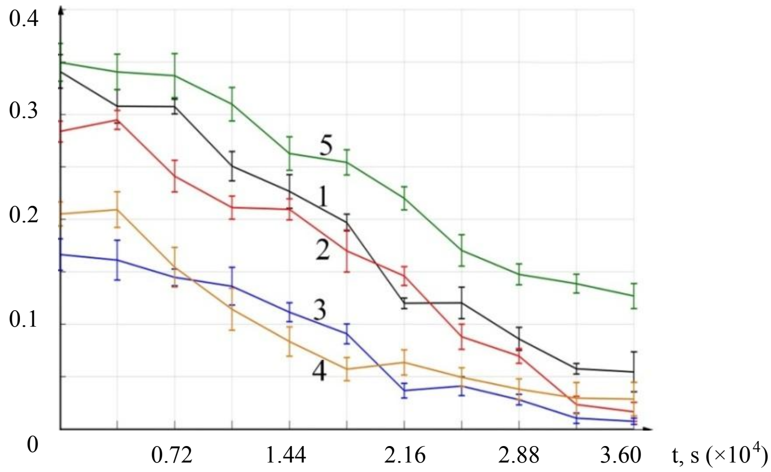

Fig. 1. Friction factor evolution in the brass-aqueous solution of hexanoic acid-steel system with acid concentration:
1 – 0.025 mol/L, 2 – 0.05 mol/L, 3 –0.1 mol/L, 4 – 0.2 mol/L, 5 – 0.5 mol/L

Comparison of the roughness parameters of the formed antifriction copper film under surface scanning by optical profilometry establishes significant differences from the original topography. As a result of the frictional interaction of the brass – steel friction pair in a hexanoic acid solution, a decrease in roughness up to R_a equal to 69.4 nm was established (Fig. 3), compared to the roughness of the initial friction surface R_a equal to 118 nm (Fig. 2).

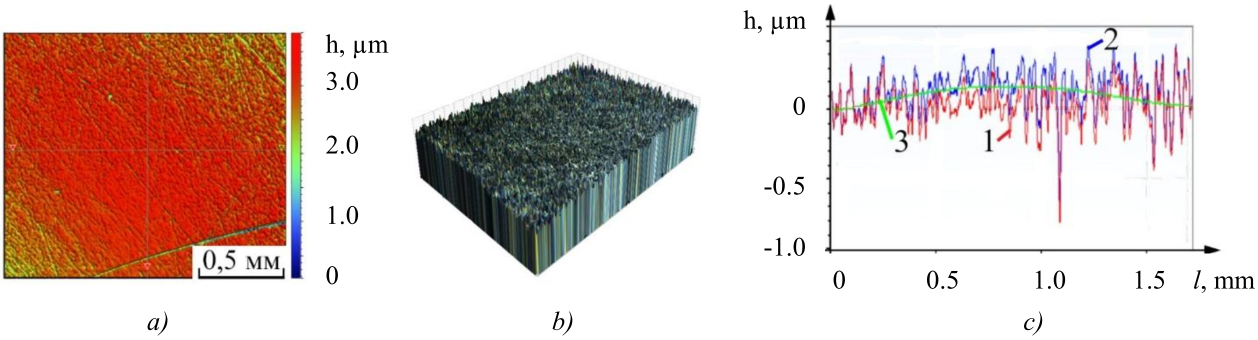

Fig. 2. Study results of the steel surface before friction by optical profilometry: *a*) 2D visualization, *b*) 3D visualization, *c*) surface profile. 1 — roughness profile, 2 — surface scan profile, 3 — waviness profile

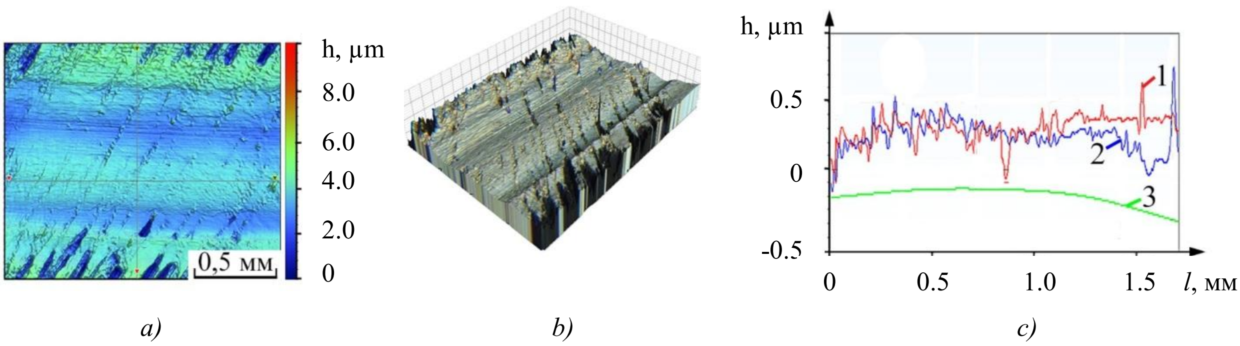

Fig. 3. Study results of the surface after friction by optical profilometry: *a*) 2D-visualization, *b*) 3D-visualization, *c*) surface profile. 1 — roughness profile, 2 — surface scan profile, 3 — waviness profile

The surface after friction is a nanochannel located parallel to the sliding direction, which is typical for the formation of a smaller amount and particle size of wear products in the lubricant [12]. This state of the friction system causes a significant decrease in the friction factor. It is known [13–15] that nanoscale irregularities, as a rule, have a lesser effect on the wear resistance of the surface than their micron counterparts due to their almost defect-free structure. In addition, the presence of nanoscale irregularities contributes to a decrease in the contact area and increases the hydrophobicity of the surface [16], which causes a decrease in the adhesion forces under friction in aqueous solutions. In this regard, nanostructured films have huge potential in the processes of decreasing the friction coefficient and protecting surfaces from wear.

Further approach to the friction surface when analysing the atomic force microscopy findings shows that the frictional interaction of a brass-steel pair in a hexanoic acid solution causes a significant modification of the friction surface as a result of the deposition of finely dispersed copper clusters that originate in the composition of the lubricating medium [17] and form a servovite film (Fig. 4).

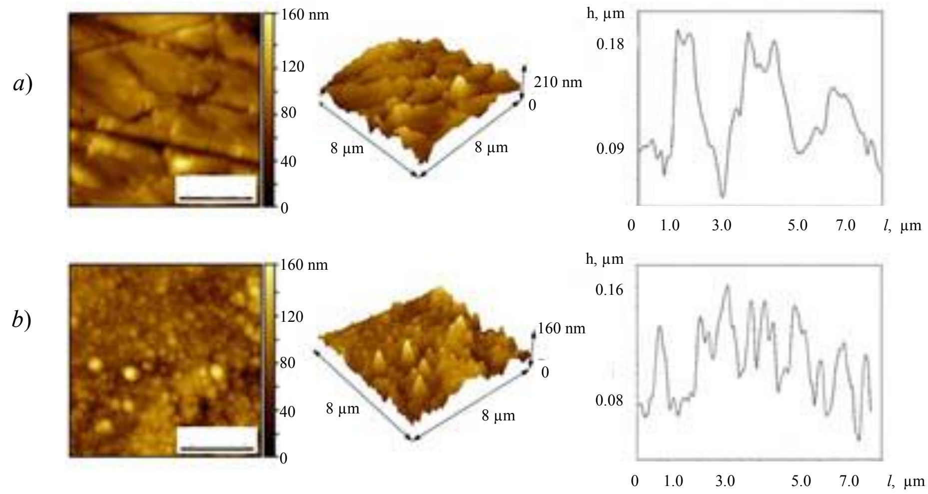

Fig. 4. Results of atomic force microscopy of the sample surface:
a) before friction, *b*) after friction of brass-steel pair in hexanoic acid solution

As a result of visualization, it is found that the steel surface after friction interaction in a hexanoic acid solution is smoothed out and get covered with fine-grained copper clusters [18, 19] due to their adsorption from the working medium composition onto the counterface (Fig. 4). At that, the layer formed on the surface is quite dense, with a small dispersion of particles in size.

To study the effect of the hexanoic acid on the anti-wear properties of a lubricant, it was instructive to consider a steel-steel friction pair. To do this, paraffinic oil was used. From 0.025 to 0.5 mass. % of hexanoic acid was introduced to its composition as an additive. The test results have shown a change in the tribological parameters of the modified oil. It was found that the dependence of the WSD ($D_и$) on the acid content in the base oil is non-monotonic in nature with a pronounced minimum at a concentration of 0.1 mass. % (Fig. 5). The addition of hexanoic acid to the lubricating composition as a modifying additive provides the least wear of the tribocouple at an acid concentration of 0.1 mass. %. In this case, the WSD decreases to 0.497 mm, while the WSD under friction of the steel-steel pair in pure paraffinic oil is 0.664 mm. At a concentration of 0.025 mass. % and 0.5 mass. %, deterioration in the tribological characteristics of the friction pair is observed. Under friction in the base oil with the addition of caproic acid with a concentration of 0.05 mass. % and 0.2 mass. %, there is only a slight decrease in the WSD (Fig. 5).

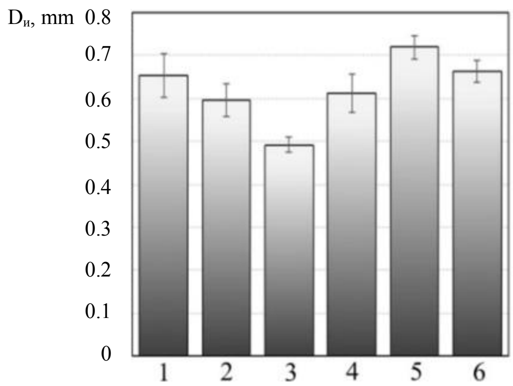

Fig. 5. Wear scar diameter dependence (D_i) on concentration of hexanoic acid in paraffinic composition:
1 - 0.025%, 2 - 0.05%, 3 - 0.1%, 3 - 0.2%, 5 - 0.5%,

The modified oil film strength analysis by the load capacity of the lubricating composition in comparison to the base oil established a change in the ultimate bearing capacity of the lubricant. When rubbing in paraffinic oil with the addition of hexanoic acid with a concentration of 0.025 mass. %, there is a slight increase in the critical load in comparison to pure base oil (Fig. 6).

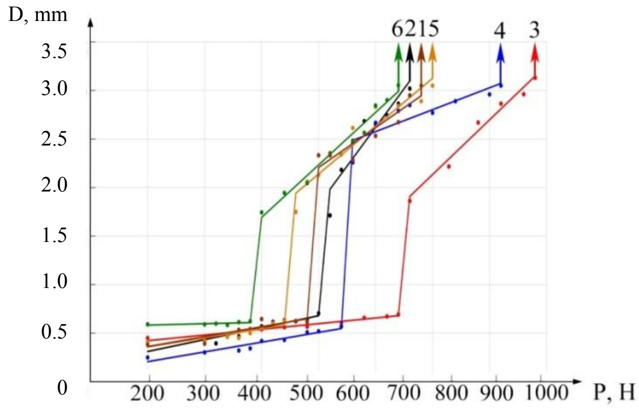

Fig. 6. Bearing and ultimate load capacity of paraffinic oil modified with hexanoic acid of various concentrations (C):
1 — pure paraffinic oil (PO), 2 — PO + 0.025 mass %, 3 — PO + 0.05 mass %, 4 — PO + 0.1 mass %,
5 — PO + 0.2 mass %, 6 — PO + 0.5 mass %

When the critical load is reached, the rubbing surfaces of the steel-steel pair get hot, the adsorption film formed in the base oil destroys, friction is enhanced, and the metal surfaces are welded at the points of contact (Fig. 6). Hexanoic acid introduced into the base oil reacts with the steel friction surface forming a more resistant chemisorption film on the protrusions of the contacting surfaces, which protects the surfaces from wear and reduces friction under conditions of high temperature and pressure, due to which the friction surfaces are smoothed out, and wear is reduced.

The best result under the steel-steel pair friction is observed in paraffinic oil modified by hexanoic acid with concentrations of 0.05 and 0.1 mass. %; in this case, the critical load ($P_к$) increases by 32%, the welding load (P_c) increases by 27% (Fig. 5). A further increase in the concentration of hexanoic acid to 0.2 mass. % and 0.5 mass. % in the lubricant composition already negatively affects its bearing and ultimate load capacity.

Discussion and Conclusions. The results obtained afford drawing the following conclusions:

- The tribological studies of a brass-steel friction pair in the hexanoic acid solution showed that the optimal molar concentration of acid in the composition of the lubricant is 0.1 mol/L.
- Under the frictional interaction of a brass-steel pair in the hexanoic acid solution, an antifriction copper film is formed on the friction surfaces, which contributes to a sharp decrease in the friction factor to 0.007 and metal wear of the friction pair up to 25 times.
- As a result of the frictional interaction of the brass-steel friction pair in the hexanoic acid solution, a decrease in roughness was established, compared to the initial friction surface.
- It was found that the frictional interaction of a brass-steel pair in the hexanoic acid solution causes a significant modification of the friction surface as a result of the deposition of finely dispersed copper clusters formed in the lubricating medium composition and forming a servovite film.
- It was found that the WSD dependence on the acid content in the base oil is non-monotonic in nature with a pronounced minimum at a concentration of 0.1 mass. %
- It is shown that the addition of 0.1 mass. % of hexanoic acid in the lubricant composition exhibits the smallest wear of the steel-steel tribopair, the WSD decreases to 0.497 mm, the critical load ($P_к$) and weld load (P_c) increase by 32% and 27%, respectively.

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Authors:

Burlakova, Victoria E.,
Head of the Chemistry Department, Don State Technical University (1, Gagarin Sq., Rostov-on-Don, 344000, RF), Dr.Sci. (Eng.), professor,
ORCID:http://orcid.org/0000-0003-3779-7079
vburlakova@donstu.ru

Drogan, Ekaterina G.,
senior lecturer of the Chemistry Department, Don State Technical University (1, Gagarin Sq., Rostov-on-Don, 344000, RF),
ORCID: http://orcid.org/0000-0002-4002-2082
ekaterina.drogan@gmail.com