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# Mechanical properties of servovite films formed in aqueous solutions of carboxylic acids under friction\*

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Механические свойства сервовитных пленок, формирующихся при трении в водных растворах карбоновых кислот\*\*\*

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Introduction. The effect of the organic component nature in the systematic series of monocarboxylic acids on the tribological characteristics of the brass-steel friction pair in aqueous solutions is described. Dependence of the mechanical-and-physical properties of the antifriction films formed during friction on the nature of the lubricating composition is investigated. The work objectives are to study the applicability of carboxylic acids as an antifriction lubricant component; to assess their effect on the mechanical properties of the servovite film formed under the brass – steel friction.

Materials and Methods. Tribological studies of the brass-steel friction pair on the AE-5 end-type friction machine are carried out. Roughness parameters of the servovite film were determined through the optical profilometry. The microgeometry and the object structure at the nanoscale were considered using atomic force microscopy. The mechanical characteristics of the antifriction film were investigated using the instrument nanoindentation.

Research Results. Tribological characteristics of the brass-steel tribocoupling and mechanical-and-physical properties of the servovite film formed during friction in the "brass – aqueous solution of carboxylic acid – steel" system were studied. It is established that the friction factor reduces when increasing the hydrocarbon radical length. The dimensional effects are found in the mechanical and tribological properties of the servovite film formed on the surface of the friction interaction in the carboxylic acids.

Discussion and Conclusions. The study results show that the

Введение. В работе показано, каким образом природа органической компоненты в систематическом ряду одноосновных карбоновых кислот влияет на трибологические характеристики пары трения «латунь — сталь» в водных растворах. Изучена зависимость физико-механических свойств антифрикционных пленок, формирующихся при трении, от природы смазочной композиции. Цели работы: изучить возможности использования карбоновых кислот как антифрикционных компонентов смазочного материала; оценить их влияние на механические свойства сервовитной пленки, формирующейся при трении латуни по стали.

Материалы и методы. Проведены трибологические исследования пары трения «латунь — сталь» на машине трения торцевого типа АЕ-5. Параметры шероховатости сервовитной пленки определялись с помощью оптической профилометрии. Микрогеометрия и структура объекта на наноуровне исследовались с помощью атомно-силовой микроскопии. Механические характеристики антифрикционной пленки изучали с помощью инструментального наноиндентирования.

Результаты исследования. Изучены трибологические характеристики трибосопряжения «латунь — сталь» и физико-механические характеристики сервовитной пленки, формирующейся при трении в системе «латунь — водный раствор карбоновой кислоты — сталь». Установлено, что при увеличении длины углеводородного радикала коэффициент трения снижается. Обнаружены размерные эффекты в механических и трибологических свойствах сервовитной пленки, формирующейся на поверхности фрикционного взаимодействия в водных растворах карбоновых кислот.

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friction interaction on the wearing surface in the aqueous solutions of carboxylic acids forms a nanostructured servovite film which drops the friction factor. Its mechanical, physical and tribological parameters depend on the composition of the model lubricating medium. It is determined that the local mechanical-and-physical properties depend on the method of producing the servovite layer, the load and the size of the deformation zone. The results obtained can be used in the development of lubricants.

**Keywords**: friction factor, selective transfer, servovite film, dimensional effects, surface roughness.

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Обсуждение и заключения. Результаты исследования показывают, что при фрикционном взаимодействии на поверхности трения в водных растворах карбоновых кислот формируется наноструктурная сервовитная пленка, резко снижающая коэффициент трения. Ee физикомеханические и трибологические параметры зависят от состава модельной смазочной среды. Определено, что локальные физико-механические свойства зависят от способа получения сервовитного слоя, нагрузки и размера зоны деформирования. Полученные результаты могут быть использованы при разработке смазочных материалов.

**Ключевые слова:** коэффициент трения, избирательный перенос, сервовитная пленка, размерные эффекты, шероховатость поверхности.

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**Introduction.** A rapid development of modern machine building increases opportunities of constructing machines that operate under the extreme conditions. Thus, the motivation of the life growth of the friction units rises with the demanding requirements for the mechanical properties of materials.

The friction conditions with high energy density impose a number of specific requirements on the machines and equipment in terms of the tribological characteristics, reliability and service life. The critical working conditions of tribo-mating surfaces affect the flow of force, loss of energy, and, eventually, dynamic behavior of the complete mechanism. Space distributions of topographic irregularities, as a rule, negatively affect the operational capabilities of machines and mechanisms. In other words, the load-bearing capacity of friction units hinges on the basic parameters of the surface state. They affect the interaction conditions in the contact area, adhesion, and deformation, thereby generating vibrations during the friction process [1] and causing uneven wear.

In this case, the greatest antifriction efficiency is demonstrated by the lubricants [2-6] containing metal additives, such as copper, aluminum, silver. These materials form protective metal plaque shells on the tribo-mating surfaces in the process of friction. Thus, a low friction factor and a medium low wear are provided in a wide range of contact pressures and slip velocities [2, 3]. The process of metal plaque filming of the surface layers in-service enables to "heal" surface defects (cracks, pores, accumulated fatigue damage). This essentially increases the antifriction characteristics and improves the mechanical-and-physical properties, which is of prime importance for the reliable operation of the friction units, and the lifetime extension.

It is notable that the formation of transfer films is possible under friction in aqueous-alcoholic media with no visible total wear of the mating surfaces [7, 8]. In the engineering practice, the use of selective transfer gives a real opportunity for designing long-life and effective friction units in the movable mating of parts and components of machines and mechanisms [8].

In this context, it is interesting to consider the effect of the lubricating medium composition on the mechanical and tribological parameters of the servovite film forming in the "brass-steel" tribo-pair (aqueous solutions of carboxylic acids are used as a lubricant medium).

**Materials and Methods.** Evolution of the friction factor of the "brass 59 – aqueous solution of carboxylic acid – steel 40X" system was investigated in the "Hybrid Functional Graphene-Based Materials" laboratory of the Research and Education Center (REC) "Materials" on the AE-5 end-type friction machine. The experiments were carried out under the following conditions: rotation speed was 180 rpm; axleload was 98 N; the test time was 10 hours. The

monocarboxylic acids of the limiting series with the general formula of R - COOH ( $R = C_n H_{2n+1}$ ) were used as an organic component of the lubricant composition.

The mechanical characteristics (hardness – H and Young modulus – E) of servovite films were determined by the instrument indentation method [9, 10]. Thereat, we used:

- -Nanotest Platform 3 (Micromaterials, UK) nanoindentation tester with the attached function unit that enables to impose loads from 0.01 mN to 500 mN;
- Nanotest Platform 3 (Micromaterials, UK) tribological nanoindentation tester, a multifunctional system for mechanical testing of materials through the dynamic nanoindentation.

The nanoindentation technique makes it possible to study the package of mechanical properties of light boundary layers of solids and films up to a few tens of nm thick [9-15]. The mechanical characteristics were studied using a diamond Berkovich indenter. Taking into account the specific thickness of the investigated films (from several hundred nanometers to units of micrometers), the operational mechanical parameters were measured at the nanoscale at indentation depths from 20 nm to the micron units. At the nanoscale, the friction coefficient was investigated on the *Triboindenter* TI-950 with simultaneous application of normal and lateral loads to the indenter [11, 16, 17]. The normal and lateral components of the forces ( $F_N$  and  $F_L$ ) and displacements ( $h_N$  and  $h_L$ ) implemented during the replication of the tribo contacts by the indenter were recorded continuously. The recorded data were analyzed, which permitted to describe the friction and wear processes. The friction coefficient  $k_{mp}$  was an evaluation item.

The *Contour GT-K1* optical profilometer with *Vision 64* analytical software installed in the Collective Resource Center of Research and Education Center "Materials" (http://nano.donstu.ru/) was used to determine the servovite film thickness and roughness parameters. The measurements were taken using vertical scanning interferometry (*VSI*) at the scan rate of  $0.1 \, \mu m$  / s with the repeatability of *RMS*  $0.01 \, nm$ .

The servovite film surface topography was studied using the *PHYWE Compact* atomic-force microscope (AFM) in a semi-contact mode with a single-crystal silicon aluminum-coated probe.

Research Results. A servovite film, spontaneously arising under the friction interaction in the glycerin solution in the copper-steel alloy, is studied fairly well [8, 18]. It is common knowledge that it is formed on the friction face under the topographically unequal conditions. Thus, in different places of frictional contact, the properties of the servovite film are different. Its thickness is less than 2  $\mu$ m, and the mechanical, physical and tribological properties differ from those of base compact copper.

It should be mentioned that one of the glycerin tribooxidation [19] products is carboxylic acid. In that context, it is interesting to consider the effect of the lubricating medium composition on the mechanical and tribological properties of the "brass-steel" tribo-pair under friction in the systematic series of monocarboxylic acids.

Long-term evolutionary tribological studies of the "brass-steel" friction pair in aqueous solutions of carboxylic acids have revealed the following dependence of the tribological characteristics on the lubricating medium composition: the length of the hydrocarbon chain of the acid radical increases from  $C_0$  to  $C_{5}$ , and the friction factor decreases to 0.007.

At that, the lowest value of the friction factor, which is characteristic for the systems implementing the wear-lessness effect [20, 21], is obtained in aqueous solutions of the valeric and caproic acids. At the same time, the "brass-steel" tribo-pair wear is reduced by 25 times. As a result of the selective transfer under the tribo-mating surfaces friction, a copper film with different roughness and surface coating density is formed.

The friction surface was scanned under transition conditions in the series of the "formic - acetic - propionic - oil - valeric - caproic" acids. At that, optical profilometry has revealed, first, a decrease in the servovite film roughness. Secondly, the dependence of roughness on the initial topography of the of the test steel disc surface (its  $R_a$  is equal to 118 nm) and on the lubricant composition was determined [22, 23] (Fig. 1).

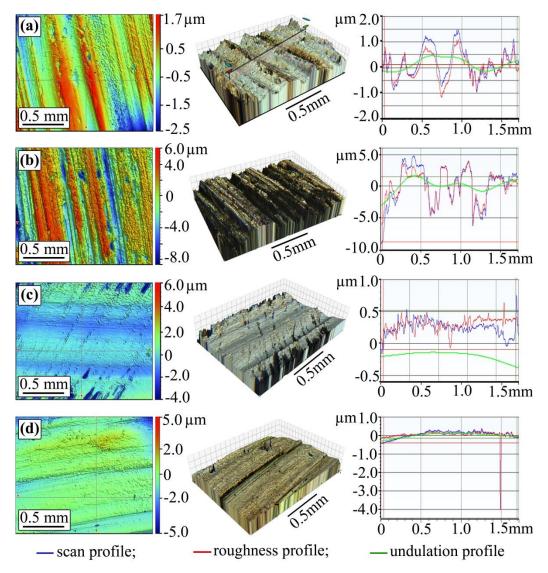


Fig. 1. 2D visualization, 3D visualization and surface profile from steel (a), (c) and brass-59 (b), (d) after friction in aqueous solutions of acetic (a), (b) and caproic (c), (d) acids

Roughness  $R_a$  at the base length ( $L = 800 \mu m$ ) of the servovite film decreases, for example, in the aqueous caproic acid, up to 69 nm. (For comparison: this index comes up to 580 nm in an aqueous solution of formic acid and to 401 nm – in an acetic acid solution.) The difference is due to the corrosive activity of the medium.

In the formic and acetic acid solutions, scores of scratches, irregularities and pores occur on the tribo-mating surfaces, as well as areas with a copper film formed in the island growth mode. In the cross-section of the friction surface, deep longitudinal grooves are revealed, which goes to show the abrasive wear pattern of the tribocoupling [23, 24].

A more detailed scanning of the surface using the AFM method and image visualization discovers considerable damage under friction in the formic and acetic acid solutions. On a 3D image, they are visualized as macroscopic dark bands corresponding to the areas of intense frictional stress accompanied by strong abrasive wear of tribo-combinations (Fig. 2).

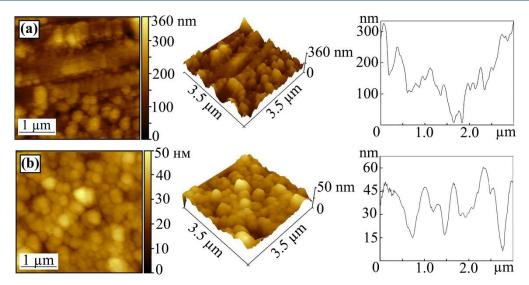


Fig. 2. Topography of servovite film surface obtained under friction in aqueous solution of acetic (a) and caproic (b) acid

A comparative analysis of the friction surface morphology upon transition to the acids with a longer hydrocarbon radical, suggests the wear pattern transition, and the structural modification of a thin subsurface film of tribocoupling.

It follows from the mass transfer of the components of the contacting bodies, as well as from the adsorption of copper nanoclusters from the working medium to the counterbody as a result of tribo-electrochemical processes in the friction zone during the implementation of the selective transfer. At this, a copper film of fine-grained copper nanoclusters is formed on the steel surface under the friction interaction in the aqueous solutions of caproic and valeric acids. The layer formed on the surface is fairly dense, with a small spread of particles in size (see Fig. 1, 2).

By now, a significant amount of experimental data has been accumulated [25-29] implying a major change in the mechanical properties with reducing the characteristic dimensions of the elements of the object structure of less than 100 nm. This permits to assume a change in the mechanical parameters of the servovite films as compared to those for copper in the volume [30]. To identify this fact, an instrument indentation was carried out.

The hardness values of H and Young's modulus E were determined by the Oliver-Pharr method [10] from the characteristic P-h diagrams (Fig. 3) in accordance with the standards [9].

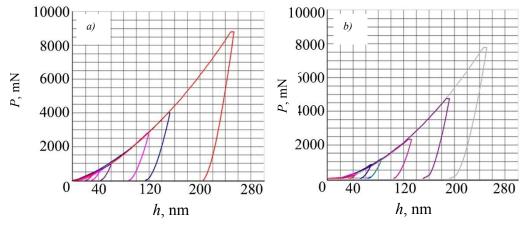


Fig. 3. Representative *P-h* diagrams for servovite film obtained through friction of "brass-steel" pair in aqueous solution of valeric acid (*a*), caproic acid (*b*)

The undertaken studies showed an increase in the values of H and E films compared to the similar characteristics of the copper sample.

The increase in the hardness H and the elasticity modulus E of the film formed on the friction surface can be ascribed to the change in the plastic deformation mechanism. A high vacancy concentration at the grain boundaries during friction interaction promotes grain-boundary sliding of building blocks relative to each other. In this case, the non-dislocation plasticity mechanism is implemented [31-34], which results in improving strength characteristics of the copper surface layer, and in increasing ductility during the transition in the series of the "formic - acetic - propionic - oil - valeric - caproic" acids.

Besides, the analysis of the dependence of the H and E values of the servovite film on the depth of the  $h_c$  plastic impression shows that the hardness is affected by the scale factor. The Young's modulus values remain practically constant up to the depths associated with the servovite layer thickness. The films formed through friction in valeric acid are of much evidence (Fig. 4).

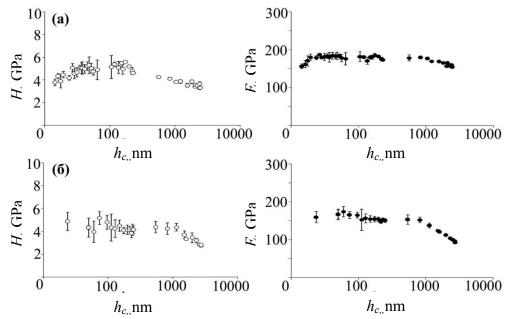


Fig. 4. Dependence of hardness (H) and Young's modulus (E) on the depth of plastic impression ( $h_c$ ) (shown in semilog coordinates) when indenting servovite film obtained in aqueous solution of valeric acid (a), caproic acid (b)

The following dimensional effect is identified:

- H value increases with growth of  $h_c$  in the shallow depth-region;
- with further growth of  $h_c$ , H falls dramatically.

For other samples, the dimensional effects are feebly-marked. Thus, for example, for the film formed in caproic acid, the values of H and E remain practically constant within the full tested range of  $h_c$  (from 23 nm to 1.1  $\mu$ m). Then, with increasing  $h_c$ , the values of H and E decrease. Obviously, this is due to the increase in the deformation zone size: the properties of the transition layer and the substrate material start to affect H and E. And, in the region of deep depths of the indentation ( $h_c > 1.1 \mu$ m), the values of H and E decrease (see Fig. 4).

The dimensional effect [35, 36] is also found under studying the dependence of the friction factor on the applied load in the nano- and microscale [11, 16, 17] (Fig. 5).

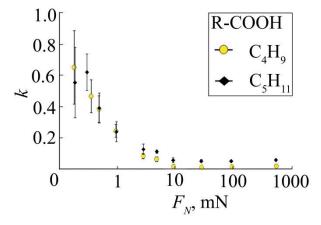


Fig. 5. Dependence of friction factor k of servovite film on load in nanoscale

First, it is expressed in a sharp drop in the friction coefficient with an increase in the normal load, up to some value of  $F_{Nonm}$ . Then, the friction coefficient increases smoothly with increasing  $F_N$ . So, the dependences  $k = f(F_N)$  have sharp minimum if some value of  $F_{Nonm}$  is reached [11, 16, 17].

The experimental data obtained can be compared to the theoretical dependence of the friction factor on the normal load. Hence, it is becoming apparent that they are in good qualitative agreement to all the film samples under

study. The  $F_{Nonm}$  value is affected by the type of carboxylic acid used as a lubricant. For example, for caproic acid,  $F_{Nonm} = 30 \text{ mN}$ , and for valerian acid,  $F_{Nonm} = 100 \text{ mN}$ .

**Discussion and Conclusions.** The mechanical properties of servovite films formed in the "brass-steel" friction pair are studied. The dimensional effects in the mechanical (hardness) and tribological (friction factor) properties are identified in the nano- and microscale. It is shown that the properties studied and the nature of their changes depend on the type of carboxylic acid used. The decrease in the average size of crystallites in the composition of film nanoparticles leads to an increase in its strength and the development of superplasticity under tension and shear.

The results obtained in the paper permit to draw the following conclusions.

- 1. During the friction interaction of the "brass-steel" pair in aqueous solutions of carboxylic acids, an antifriction servovite film is formed on the friction surfaces, which contributes to a sharp decrease in the friction factor.
  - 2. The servovite film, which is formed under the tribo-interaction of the "brass-steel" pair, is nanostructured.
- 3. The tribological parameters of the "brass aqueous carboxylic acid steel" system depend on the length of the hydrocarbon radical of carboxylic acid used as a lubricant.
- 4. The lubricating medium composition affects the roughness parameters of the servovite film: in the transition from formic to caproic acid, the film surface roughness is reduced to one-quarter.
- 5. Local physical and mechanical properties depend on the conditions for obtaining the servovite layer, the load, and the size of the deformation zone (the indentation depth).
- 6. The detected dimensional effects (dependence of the determined values on the indentation depth) verify the conclusion on the nanostructured character of the servovite film formed in the "brass aqueous carboxylic acid steel" tribosystem.

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