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*O.V. Stovpnyk, V.I. Sytar***INVESTIGATION OF TRIBOLOGICAL CHARACTERISTICS OF POLYMER COATING BASED ON PHENYLONE MODIFIED BY COMPLEX COPPER (II) COMPOUNDS OF THE COMPOSITION $[\text{Cu}(\text{HL})\text{X}_2]_2$** **Ukrainian State University of Science and Technologies, Dnipro, Ukraine**

Antifriction coatings based on aromatic phenylene polyamide (phenylone) were developed by introducing a copper arylamide complex with benzimidazole-2-thiocarboxylic acid as a modifying additive. The tribological properties of the phenylene-modifying additive system were investigated, focusing on the nature and concentration of the modifying additive in the polymer composition. The influence of the additive on the microhardness of the polymer coatings was assessed. The introduction of the copper arylamide complex with benzimidazole-2-thiocarboxylic acid as a modifying additive resulted in changes to the polymer matrix structure, forming strong chemical bonds between polymer chains, which improved the strength of materials and reduced its wear susceptibility. The tribological properties of the modified coatings were characterized by a lower coefficient of friction and better wear resistance compared to unmodified coatings. The developed coatings can be widely applied in various industries, such as mechanical engineering, aviation, and automotive, offering benefits such as enhanced durability, wear protection, and reduced maintenance costs. Additionally, their use can contribute to lower energy consumption and reduced environmental impact.

Keywords: friction, wear resistance, phenylone, thioamide, modifier, microhardness.

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Introduction

Friction units play an important role in many mechanical systems, where the use of efficient materials can significantly improve the performance and durability of equipment. In recent decades, new polymeric materials have been actively researched to improve the properties and performance of friction units. One such promising material is phenylone [1,2].

Friction mating applications operating under high load and temperature conditions present a challenge to many engineers and designers. Polymeric materials, despite their good wear resistance properties, typically have limitations in terms of thermal conductivity and heat resistance, which can limit their use in such environments. To address this problem, systems incorporating friction interfaces with polymer coatings on heat resistant metal substrates are being developed.

This makes it possible to combine the advantages of polymers and metals, creating reliable and heat-resistant friction systems [3].

Many factors play a key role in the design and optimization of friction interfaces with polymer coatings, but one of the most important is the composition of the polymer composition. The correct choice of the coating composition can significantly influence its friction characteristics and the overall performance of the friction unit. The most promising element of tribological unit is polymer coating [4].

The use of polymer coatings as antifriction materials provides a number of significant advantages that can significantly improve the operation of friction units and mechanisms [5,6]. The main advantages and prerequisites for the application of these technical solutions are as follows:

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Investigation of tribological characteristics of polymer coating based on phenylone modified by complex copper (II) compounds of the composition $[\text{Cu}(\text{HL})\text{X}_2]_2$

– stress equalization: polymer coatings, like lubricants, can equalize stresses in the friction zone, resulting in lower specific pressures and therefore less wear on the surfaces. This is particularly important under high load conditions;

– reduction of potential surface interaction: the low thermal conductivity of polymeric materials in the friction zone creates a temperature gradient that reduces potential interaction between the surface layers of the materials, thus reducing wear and damage;

– increased rigidity and damping capacity: polymer coatings have high rigidity and the ability to absorb vibration and shock, which helps to reduce the mechanical component of the friction force and prevent damage to surfaces;

– improved thermal conductivity: a thin layer of polymer can significantly improve thermal conductivity in the friction zone, which promotes more uniform heat distribution and reduces thermal deformation;

– adhesion strength: it is important to ensure a sufficient level of adhesion strength between the polymer coating and the substrate to avoid loosening of the tension when inserting the polymer bushings into the metal shells. This ensures a secure connection and prevents defects in the friction unit.

Overall, the use of polymer coatings as sliding materials provides a complete solution for optimizing the performance, wear resistance and productivity of friction assemblies, especially under high load and temperature conditions.

Phenylone as an antifriction material offers some significant advantages, especially in heavily loaded friction units at elevated temperatures. Its high wear resistance and ability to operate under significant loads and velocities make it a valuable material for many industrial friction units, which allows its use in heavily loaded friction units, namely its ability to operate under loads of up to 20 MPa and sliding velocities of up to 2–3 m/s [7].

However, the disadvantages of phenylone must also be considered, namely the relatively high coefficient of friction, which can lead to overheating and jamming of the friction unit, especially when operating in dry conditions. In most cases, phenylone requires lubricants for optimum performance, which can increase maintenance and operating costs.

The outcome of the choice of phenylone in friction assemblies should be justified by a careful analysis of the requirements of the specific application and an evaluation of the balance between its advantages and disadvantages.

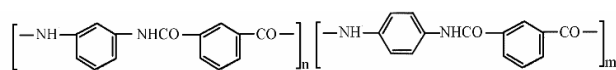
To overcome the disadvantages of phenylone described above, a large number of composites have

been developed on its basis. The following materials have been used as fillers of aromatic polyamide: graphite, molybdenum disulfide, boron nitride, talc, organosiloxanes, organic and inorganic fibers, fluoroplastic, silicon- yttrium oxynitride, metal oxides and metals in pure form. All of these fillers were used to selectively enhance a particular property of phenylone, and in most cases, other properties of the material were degraded. To improve the antifriction properties of phenylone, solid layered lubricating materials are introduced into its composition: graphite, molybdenum disulfide, boron nitride, talc, in different polymer-filler ratios [8–10].

This article presents the results of the research of a problem related to the study of regularities of phenylone modification by complex compounds of copper (II) with heterocyclic thioamide ligands in order to increase tribological properties of polymer coatings.

Experimental

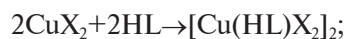
Phenylone C2 is a linear heterochain copolymer containing in the main chain of the macromolecule an amide group —HNCO— connected on both sides by phenyl fragments is obtained by emulsion polycondensation [11,12] in the system of terephthalic acid dichlorohydride with a mixture of p- and m-phenylenediamine taken in equimolar ratios:

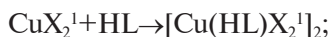


Phenylone C2 is a fine powder (20–200 μm) with a bulk density of 0.33 g/cm^3 , prepared by heterophase polycondensation of p-phenylenediamine and isophthalic acid; it has an average molecular viscosity of 20000–70000 (light scattering method).

Phenylone after synthesis has an amorphous structure with glass transition temperature $T_g=563\text{ K}$; but in the temperature range of 613–633 K, it rapidly crystallizes. Phenylone has a sufficiently high (up to 533 K) long-term operation temperature, increased radiation and chemical resistance and other useful properties.

The copper complex compounds derivatives of benzimidazole-2-thiocarboxylic acid arylamides are proposed as coating modifiers in this study. Heterocyclic thioamides are known [13] to form complex compounds with copper (II) salts of the composition $\text{Cu}(\text{HL})\text{X}_2$. Benzimidazole-2-thiocarboxylic acid arylamides also readily form the above-mentioned $[\text{Cu}(\text{HL})\text{X}_2]_2$ complex compounds according to the following scheme:

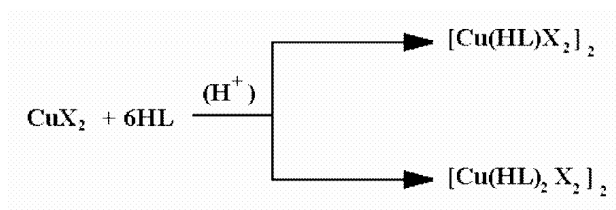




where $\text{X} = \text{ClO}_4, \text{NO}_3, \text{Cl}, \text{Br}$; $\text{X}_1 = \text{C}_6\text{H}_5\text{SO}_3, 4\text{-CH}_3\text{C}_6\text{H}_4\text{SO}_3$.

The reaction proceeds in anhydrous medium under heating, usually in the presence of the acid of the same name HX . The duration of the interaction and other features depend on the nature of the anion X^- and the structure of the thioamide ligand.

In case of synthesis of complex compounds $\text{Cu}(\text{CHAl}_3\text{COO})_2$ (where $\text{Hal} = \text{Cl}, \text{F}$) with HL , the reaction proceeds with formation of a mixture of complexes of two forms: $[\text{Cu}(\text{HL})\text{X}_2]$ and $[\text{Cu}(\text{HL})\text{X}_2]_2$ according to the following scheme:



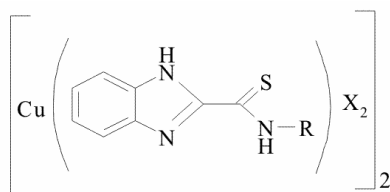
where $\text{X} = \text{CCl}_3\text{COO}, \text{CF}_3\text{COO}$.

Then the mixture of compounds is separated. In the case of interaction of CuY salts with HL in Solv solvent, the obtained $\text{Cu}(\text{II})$ complexes contain a coordinated Solv solvent molecule.

The composition of the synthesized complexes was proved by elemental analysis, and the structure was proved by IR-, ESR- spectroscopy, and X-ray diffraction analysis. In particular, the dimericity of the complexes $[\text{Cu}(\text{HL})\text{X}_2]_2$ was confirmed by their diamagnetization, and the monomericity of $[\text{Cu}(\text{HL})\text{X}_2]_2$ was confirmed by the presence of the characteristic signal of the paramagnetic cation Cu^{+2} and $[\text{Cu}(\text{HL})(\text{Solv})\text{Y}]$ in the ESR spectrum of these substances.

As initial materials for research of antifriction properties, we used aromatic polyamide phenylone C2, serving as a binder, porous bronze with an ordered depth of pores, as a substrate, and complex compounds of copper (II) derivatives of arylamides benzimidazole-2-thiocarboxylic acid, acting as modifiers.

Copper (II) complex compounds were synthesized according to the procedure disclosed in more detail in ref. [14], they can be represented by the following structural formula



where R is the substituent of the thioamide ligand; and X is the anion of the complex compound.

The obtained compounds are substances of green, light brown, and yellow-green colors, insoluble in alcohols (precipitating after the process), hot water and even more so they are hydrolyzed in alkali solutions.

Previously, compounds of similar structure have shown their effectiveness as antifriction and anti-wear additives for industrial oils [14].

To determine the optimal content of the modifying additive, as well as establish the effect of its concentration on the antifriction properties of compositions, the tribological studies were carried out for compounds in which the thioamide ligand substituent and the anion complex were varied. The structural formulae of the studied compounds are given in Table.

Friction and wear tests were carried out on a friction machine SMT-2010 under the scheme of pad-disk, in liquid mode. Industrial oil Arow Wolf 8301872 (ISO 6743-4:2015) was used as a lubricant. The disk with a diameter of 50 mm was made of steel 45 (Ukrainian state standard DSTU 7809:2015) and subjected to heat treatment (45-50 HRC, $R_a = 0.63\text{--}0.4\text{ }\mu\text{m}$).

The preparation of test specimens for sliding coating tests includes the following steps:

- dissolution of phenylone and modifying additive in DMFA;
- cleaning and degreasing of the substrate in DMFA;
- placing porous bronze samples in the prepared solution and then placing them in a barometric chamber where a vacuum is created. This helps to remove air from the pores and create a vacuum in them. After the pressure is removed, the pores are filled with the solution;
- drying of impregnated samples in a desiccator at a temperature of 420 K for 1.5 h;
- the second layer of polymer coating is applied directly with a brush or by another method (e.g. dipping, spraying, vibro-vortexing, etc.);
- the sample with the applied layer is placed in a drying cabinet and kept at a temperature of 418°C for 2 hours.

More details of the sample preparation process are given in ref. [15].

Results and discussion

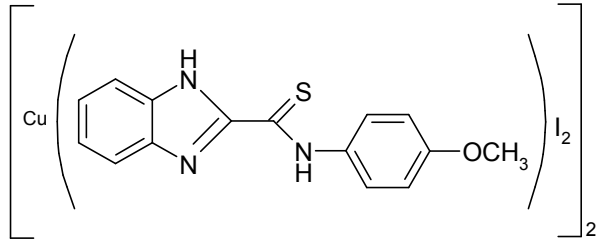
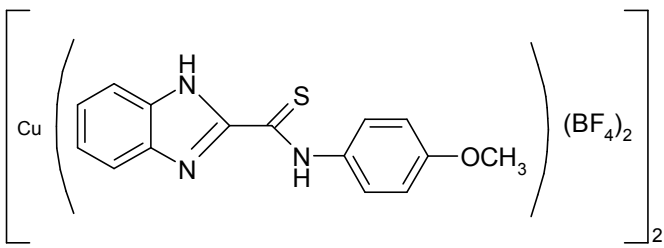
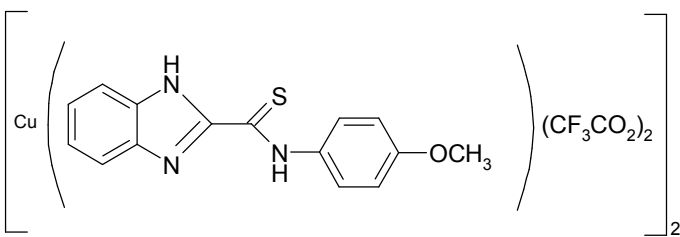
The study of tribological properties of compositions with changes in the anion atom of the complex compound and the ligand substituent was of considerable interest during the research.

The results of antifriction studies of the

Copper (II) complex compounds of the composition $[\text{Cu}(\text{HL})\text{X}_2]_2$

Designation	Chemical name	Additive formula
1	2	3
$\text{II} \cdot \text{Ar} \cdot (\text{ClO})_4$	bis-diperchlorato-bis[benzimidazol-2-N-(4-phenyl)-carbothioamide] dicopper (II)	
$\text{II} \text{ Ar} \cdot \text{Cl} \cdot \text{Cl}_2$	bis-(m-chloro)-dichloro-bis[benzimidazole-2-N-(4-chlorophenyl)-carbothioamide] dicopper (II)	
$\text{II} \text{ Ar} \cdot \text{CH}_3 \cdot \text{Cl}_2$	bis-(m-chloro)-dichloro-bis[benzimidazol-2-(N-metophenyl)-carbothioamide] dicopper (II)	
$\text{II} \text{ Ar} \cdot \text{OCH}_3 \cdot \text{Cl}_2$	bis-(m-chloro)-dichloro-bis[benzimidazol-2-(N-methoxyphenyl)-carbothioamide] dicopper (II)	
$\text{II} \text{ Ar} \cdot \text{OCH}_3 \cdot \text{Br}_2$	bis-(m-bromo)-dibromo-bis[benzimidazol-2-(N-methoxyphenyl)-carbothioamide] dicopper (II)	

Copper (II) complex compounds of the composition $[\text{Cu}(\text{HL})\text{X}_2]_2$ (Continued)

Designation	Chemical name	Additive formula
1	2	3
$\text{II Ar} \cdot \text{OCH}_3 \cdot \text{I}_2$	bis-(m-iodo)-diiodo-bis[benzimidazol-2-(N-methoxyphenyl)-carbothioamide] dicopper (II)	
$\text{II Ar} \cdot \text{OCH}_3 \cdot (\text{BF}_4)_2$	bis-(m-tetrafluoroborato)-ditetrafluoroborato-bis[benzimidazole-2-(N-methoxyphenyl)-carbothioamide] dicopper (II)	
$\text{II Ar} \cdot \text{OCH}_3 \cdot (\text{CF}_3\text{CO}_2)_2$	bis-(m-trifluoroacetato)-ditrifluoroacetato-bis[benzimidazole-2-(N-methoxyphenyl)-carbothioamide] dicopper (II)	

composition are shown in Figs. 1 and 2 under friction modes with lubrication ($P=10$ MPa and $v=0.6$ m/s). The compositions with the content of complex compounds in the range of up to 3.0% were investigated, while the polymer-additive system stratifies at higher content of modifying additive. The greatest modifying effect is achieved with the introduction of 1% of complex compound (Figs. 1 and 2): there are extremal dependences of the coefficient of friction and wear resistance. The results of the study showed that the initial phenylone has a value of friction coefficient in oil of 0.080, while these values are reduced to 0.045 with the introduction of the modifier. At the same time, the wear resistance of the modified composition increases by 60%.

Copper complex compounds have a certain effect on the friction mechanism of the composition on steel. The obtained experimental curves for antifriction properties depending on the composition of the composition are characterized by the presence of an extreme bend in the area of the greatest modifying

effect. The change of tribological properties of compositions is connected both with structural changes in the composition and with the formation of protective films on the surface of the steel counterbody.

Analyzing the obtained data on the influence of the nature of the modifier on the properties of polymer compositions, we can conclude that both the structure of the thioamide substituent and the anion of the complex compound of the modifying additive have a significant influence on the studied parameters. Thus, polymer compositions containing chlorine in the structure of the anion of the modifying additive show the best values of wear resistance and friction coefficient.

Figure 2 shows the dependence of the influence of modifying additive concentration on the friction coefficient. Analyzing the data of Fig. 1, it was found that in this series of compounds the greatest modifying effect in relation to the wear resistance of the composition is observed at the introduction of complexes containing a substituent thioamide ligand, parachlorophenyl, and anion of the complex, chlorine.

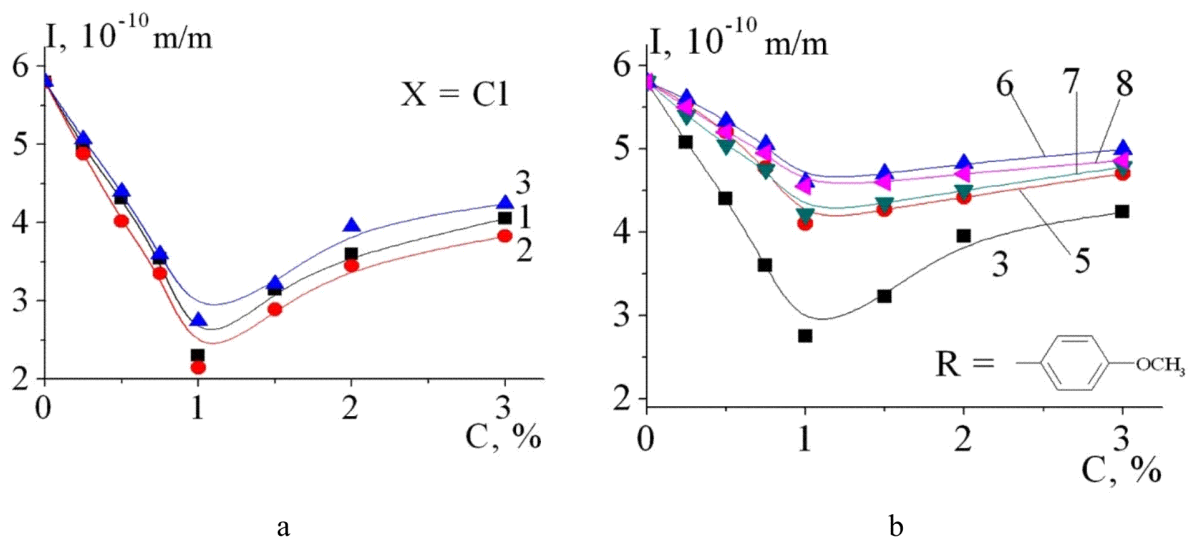


Fig. 1. Concentration dependence of wear resistance of phenylone C2+copper(II) complex compounds with the composition $[\text{Cu}(\text{HL})\text{X}_2]$: 1 – II Ar n-CH₃ Cl₂; 2 – II Ar n-Cl Cl₂; 3 – II Ar n-OCH₃ Cl₂; 4 – II Ar (ClO₄); 5 – II Ar n-OCH₃ Br₂; 6 – II Ar n-OCH₃ I₂; 7 – II Ar n-OCH₃ ((BF₄)₂); 8 – II Ar n-OCH₃·(CF₃CO₂)₂

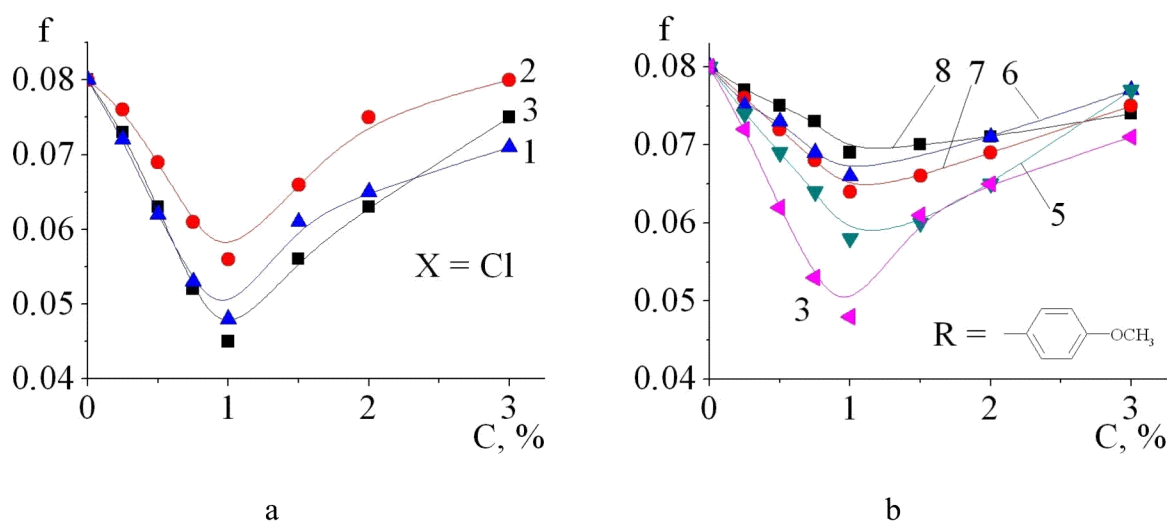


Fig. 2. Concentration dependence of the friction coefficient of the composition phenylone C2+copper(II) complex compounds with the composition $[\text{Cu}(\text{HL})\text{X}_2]$: 1 – II Ar n-CH₃ Cl₂; 2 – II Ar n-Cl Cl₂; 3 – II Ar n-OCH₃ Cl₂; 4 – II Ar (ClO₄); 5 – II Ar n-OCH₃ Br₂; 6 – II Ar n-OCH₃ I₂; 7 – II Ar n-OCH₃ ((BF₄)₂); 8 – II Ar n-OCH₃·(CF₃CO₂)₂

Based on the experimental data obtained, there is an interest in conducting comparative studies of wear resistance of optimal compositions under conditions of different levels of specific pressure in the friction unit. Analysis of the data presented in Fig. 3 demonstrates that friction conditions have a significant impact on wear resistance. As the load level increases, a systematic decrease in wear resistance is observed throughout the range considered. It is also important to note that the introduction of paramethoxyphenyl as a substituent of the thioamide ligand leads to an increase in the ultimate bearing

capacity of the friction unit.

Analysis of the data of Figs. 2 and 3 showed that the influence of the nature of the modifier also affects the value of the coefficient of friction, so the lowest values are achieved with the introduction of the composition as an anion, chlorine, as a substitute thioamide ligand, paramethoxyphenyl.

Carrying out the generalized analysis shown in Fig. 4, at a given friction coefficient of 0.06, we conclude that the highest value of antifriction properties are achieved when the composition is introduced as an anion, chlorine, as a substitute thioamide ligand,

paramethoxyphenyl.

The concentration dependences of microhardness of the developed optimal compositions were also studied in this work (Fig. 5).

After analyzing the results of research (Fig. 5), we come to the following conclusions: increasing the content of modifying additives leads to an increase in the microhardness of the composition. However, this increase in microhardness is characteristic for the concentration of additives of up to 1 wt.%, and then it begins to decrease. In particular, the introduction of complex compound II Ar n-OCH₃ Cl₂ in optimal proportions into the composition of phenylone improves its tribotechnical properties by 70%.

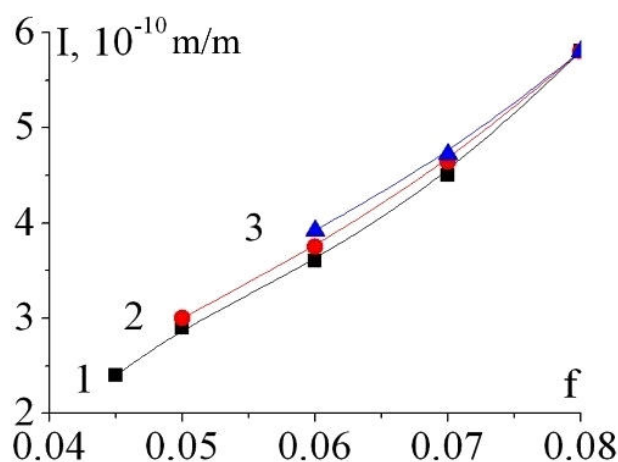


Fig. 3. Dependence of wear intensity on friction coefficient at friction with lubricant ($P_{sp}=10$ MPa and $v_{sl}=0.6$ m/s) and modifier concentration from 0 to 1% :
1 – II Ar n-OCH₃ Cl₂; 2 – II Ar n-Cl Cl₂;
3 – II Ar n-CH₃ Cl₂

In addition to studies related to the influence of complex compounds, a rather important parameter is the research related to the study of the surface of the counterbody. These studies will allow finding out the mechanism of friction in the tribological interface of parts.

Based on the analysis of the obtained data of optical microscopy (Fig. 6), it is established that large horizontal lines are observed on the friction surface before frictional interaction, which are protrusions and depressions of surface roughness. After frictional interaction with the composite, a decrease in the number and depth of these streaks is observed, which is evidence of a decrease in roughness. After friction, areas of composite transfer to the counterbody are formed due to the higher surface energy of the metal than that of the polymer in tribological interaction.

As can be seen from Fig. 6, after frictional interaction, a continuous film is formed on the surface of the counterbody. The formation of the film leads to a change in the conditions of contact between the composition and the steel counterbody and directly affects the values of the coefficient of friction and wear.

The appearance of antifriction film on the friction surface is explained by parallel processes of polymer material destruction and tribological processes occurring in the localized zone of tribological contact.

Conclusions

From the analysis of the obtained data, it can be concluded that Cu²⁺ ions as well as Cl atoms play an active role in the friction zone. These elements, as it is known [18], are often included in a variety of chemical compounds, including complex compounds, which are used as antifriction and anti-wear additives in friction units. It is especially important to note

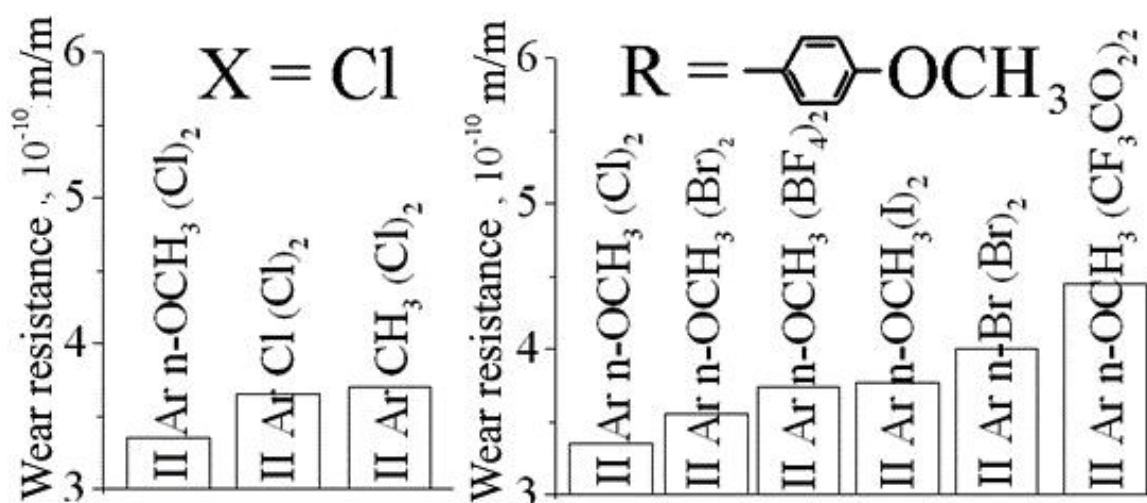


Fig. 4. Graph of comparison of wear resistance of polymer composition depending on the nature of modifying additive at a given value of friction coefficient of 0.06

that the tribological effect depends on the structure of the investigated complex compounds. As the research results show (Fig. 4), the complexes based on paramethoxyphenyl (compounds 4–8) have the lowest wear and friction coefficient indices compared to other investigated complexes of this group of compounds. According to the obtained data, it can be assumed that due to the high chelating ability of the mentioned thioamide ligands, tribochemical reactions in the polymer coating are not associated with the formation of copper sulfides and chlorides, which are usually given special importance in the process of formation of the friction boundary layer. Instead, the complexes or products of their primary interaction with the polymer play the main role. When adsorbed on the surface of the counterbody, complex compounds form a «protective» film, which contributes to the reduction

of the coefficient of friction in the friction process, due to adsorption and chemisorption on the surface of the counterbody.

It is important to note that the concentration of the modifier has a significant effect on the tribological properties of coatings. Increasing the modifier concentration leads to an increase in the inclusion size of complex compounds in the coating, which, in turn, affects the increase in wear and brittleness.

The developed antifriction coatings based on phenylone and complex compounds of copper with heterocyclic thioamide ligand can be recommended for use in friction units of machines and mechanisms operating in harsh operating conditions.

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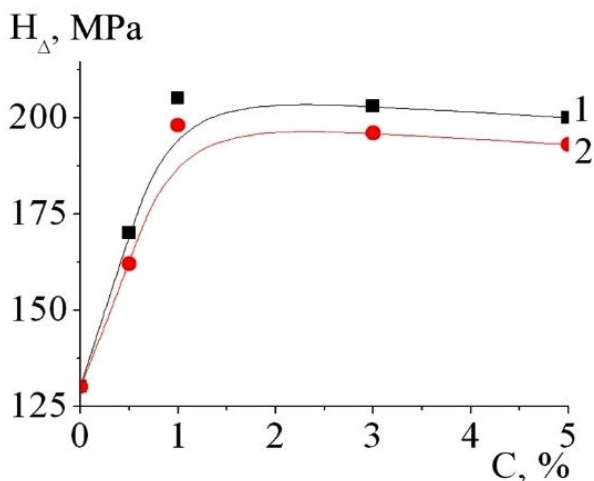
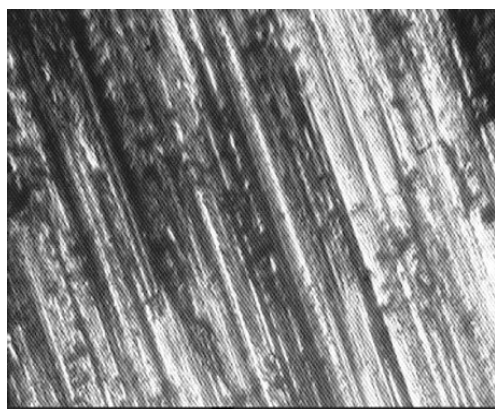
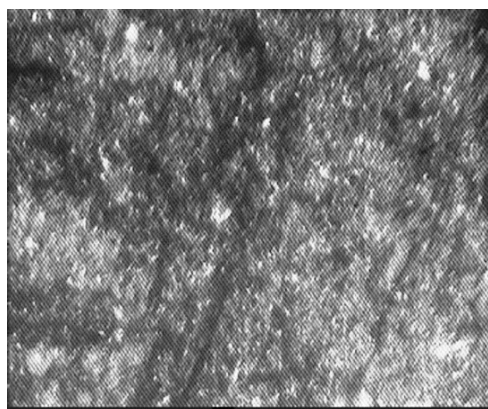


Fig. 5. Concentration dependence of microhardness of phenylone C2 composition+modifying additive:
1 – II Ar n-OCH₃ Cl₂; 2 – II Ar n-Cl Cl₂



a



b

Fig. 6. Microphotographs of the counterbody surface before (a) and after frictional interaction with phenylone C2+II Ar n-OCH₃ Cl₂ (1%) coating (b)

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ДОСЛІДЖЕННЯ ТРИБОТЕХНІЧНИХ ХАРАКТЕРИСТИК ПОЛІМЕРНОГО ПОКРИТТЯ НА ОСНОВІ ФЕНІЛОНУ, МОДИФІКОВАНОГО КОМПЛЕКСНИМИ СПОЛУКАМИ МІДІ(II) СКЛАДУ $[Cu(HL)X_2]_2$

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Розроблено та досліджено антифрикційні покриття на основі ароматичного поліаміду фенілону з використанням як модифікуючої добавки комплексу ариламід міді з бензімідазол-2-тіокарбоною кислотою. Досліджено триботехнічні властивості системи «фенілон–модифікуюча добавка» залежно від природи модифікуючої добавки та її вмісту в полімерній композиції. Досліджено вплив модифікуючої добавки на показники мікротвердості полімерних покриттів. Введення комплексу ариламід міді з бензімідазол-2-тіокарбоною кислоти як модифікуючої добавки дозволило змінити структуру полімерної матриці, утворюючи міцні хімічні зв'язки між полімерними ланцюгами. Це привело до підвищення міцності матеріалу та зниження його схильності до зношування. Триботехнічні властивості матеріалів характеризують їх поведінку при терті та зношуванні. Дослідження показали, що модифіковані покриття мають більш низький коефіцієнт тертя та знос порівняно з немодифікованими. Розроблені покриття можуть знайти широке застосування в різних галузях промисловості, таких як машинобудування, авіація, автомобілебудування. Вони можуть бути використані для захисту деталей від зношування, підвищення їх довговічності та зниження витрат на ремонт і заміну деталей. Крім того, застосування таких покриттів дозволить знизити споживання енергії та зменшити шкідливий вплив на навколишнє середовище.

Ключові слова: тертя, зносостійкість, фенілон, тіоамід, модифікатор, мікротвердість.

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INVESTIGATION OF TRIBOLOGICAL CHARACTERISTICS OF POLYMER COATING BASED ON PHENYLONE MODIFIED BY COMPLEX COPPER (II) COMPOUNDS OF THE COMPOSITION $[\text{Cu}(\text{HL})\text{X}_2]$

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Antifriction coatings based on aromatic phenylene polyamide (phenylone) were developed by introducing a copper arylamide complex with benzimidazole-2-thiocarboxylic acid as a modifying additive. The tribological properties of the phenylene-modifying additive system were investigated, focusing on the nature and concentration of the modifying additive in the polymer composition. The influence of the additive on the microhardness of the polymer coatings was assessed. The introduction of the copper arylamide complex with benzimidazole-2-thiocarboxylic acid as a modifying additive resulted in changes to the polymer matrix structure, forming strong chemical bonds between polymer chains, which improved the strength of materials and reduced its wear susceptibility. The tribological properties of the modified coatings were characterized by a lower coefficient of friction and better wear resistance compared to unmodified coatings. The developed coatings can be widely applied in various industries, such as mechanical engineering, aviation, and automotive, offering benefits such as enhanced durability, wear protection, and reduced maintenance costs. Additionally, their use can contribute to lower energy consumption and reduced environmental impact.

Keywords: friction; wear resistance; phenylone; thioamide; modifier; microhardness.

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