UDC 678.01:678.741.41:539.234

THIN-LAYER NANOCOMPOSITE COATINGS FOR CONSTRUCTION EQUIPMENT

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Abstract

Fluorine-containing coatings are promising materials for increasing the service life of equipment used in construction. The range of these compounds is quite wide, and the thickness of the coatings can vary from tens of nanometers to tens of millimeters. Interest in these coatings is due to high performance. Thin-layer coatings of fluorine-containing compounds formed from solutions were studied. These connections can be widely used in various friction assemblies of construction equipment to increase the service life. The article discusses issues related to the change in the physical and mechanical characteristics of fluorine-containing coatings, known under the trade name "Foleox." The morphology and microhardness of coatings were studied depending on the technological parameters used to create these protective layers. As surfactant, fluorine-containing oligomers of the trade name "Foleox" were used, the structural formula of which is RF – COOH (F1); RF = CONHR1 (F-AK1); RF – RF (F14) (where RF – fluorine-containing radical), RF – CONHR2 (F-AK2), B1 – water-soluble foleox. The coatings were applied by dipping the substrate in a 1–2 % solution of fluorine-containing oligomer in chladone 113 followed by air drying. A HWMMT-X7 microhardness meter was used to measure the microhardness of coatings formed on metals. The morphology was studied using an NT-206 atomic force microscope. It has been found that fluorine-containing oligomers strengthen the surface layers of the substrate if chemical (chemisorption) bonds such as salts of higher acids are formed between the film of the fluorine-containing oligomer and the metal (substrate). If the formation of a chemisorption interaction between the oligomer molecules and the substrate does not occur, plasticization of the surface layers of polycrystals occurs. Energy treatment intensifies the processes of crystal formation in thin films, as a result, the microhardness of the thin-layer composite coating increases. These coatings can be recommended for friction assemblies of construction equipment operated in severe conditions.

Keywords: nanocomposite coatings, morphology, microhardness, fluorine-containing compounds, oligomer, crystal, construction machinery.

ТОНКОСЛОЙНЫЕ НАНОКОМПОЗИТНЫЕ ПОКРЫТИЯ ДЛЯ СТРОИТЕЛЬНОЙ ТЕХНИКИ

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Реферат

Фторсодержащие покрытия являются перспективными материалами для увеличения срока службы техники, применяемой в строительстве. Ассортимент данных соединений достаточно широк, а толщина покрытий может варьироваться от десятков нанометров до десятков миллиметров. Интерес к данным покрытиям обусловлен высокими эксплуатационными характеристиками. В работе исследованы тонкослойные покрытия фторсодержащих соединений, формируемых из растворов. Данные соединения возможно широко применять в различных узлах трения строительной техники для увеличения срока эксплуатации. В статье рассмотрены вопросы, связанные с изменением физико-механических характеристик фторсодержащих покрытий, известных под торговым названием «Фолеокс». Изучена морфология и микротвердость покрытий в зависимости от технологических параметров, применявшихся при создании данных защитных слоев. В качестве ПАВ использовали фторсодержащие олигомеры с торговым названием «Фолеокс», структурная формула которых имеет вид RF – COOH (Ф1); RF – CONHR1 (Ф-АК1); RF – RF (Ф14) (где RF – фторсодержащий радикал), RF – CONHR2 (Ф-АК2), B1 – водорастворимый фолеокс. Покрытия наносили путем окунания подложки в 1–2 % раствор фторсодержащего олигомера в хладоне 113 с последующей сушкой на воздухе. Для измерения микротвердости покрытий, сформированных на металлах, использовали микротвердомер HWMMT-X7. Морфологию изучали с применением атомно-силового микроскопа NT-206. Установлено, что фторсодержащие олигомеры упрочняют поверхностные слои подложки, в случае если происходит образование химических (хемосорбционных) связей типа солей высших кислот между пленкой фторсодержащего олигомера и металла (подложкой). В случае если не происходит образование хемосорбционного взаимодействия между молекулами олигомера и подложкой происходит пластифицирование поверхностных слоев поликристаллов. Энергетическая обработка интенсифицирует процессы кристаллообразования в тонких пленках, в результате повышается микротвердость тонкослойного композиционного покрытия. Данные покрытия могут быть рекомендованы для узлов трения строительной техники, эксплуатируемой в тяжелых условиях.

Ключевые слова: нанокомпозиционные покрытия, морфология, микротвердость, фторсодержащие соединения, олигомер, кристалл, строительная техника.

Introduction

Fluorine-containing coatings are a promising material for increasing the service life of construction equipment. These materials consist of layers ranging in thickness from nanometers to several millimeters, consisting of fluorinated polymers, oligomers, or polymer-oligomer compounds. The most common representative of this class of materials polyvinylidene fluoride

https://doi.org/10.36773/1818-1112-2025-138-3-99-103

(PVDF). These coatings are known for their high performance characteristics: high chemical resistance, resistance to ultraviolet radiation, high Mechanical strength, hydrophobicity, and heat resistance. Another fluorinated material widely used in the construction industry is polytetrafluoroethylene. It has unique anti-adhesive properties. It is often used not as a decorative but as a functional coating for friction units (bearings, sliding surfaces) to reduce the adhesion of soil, clay, and concrete [1–3]. The use of coatings based on this chemical compound can significantly increase the durability and appearance of the product, provide protection against corrosion, increase resistance to mechanical damage, and reduce the adhesion of various types of contaminants to the surface of construction equipment, which improves environmental friendliness and reduces fuel consumption. Typically, the thickness of these coatings is 100 µm or more. Another area is the use of thin-film coatings with a thickness of about 0,5 µm to 10 µm. These coatings are formed on the surface of solids from solutions and using vacuum methods. Formation from solutions is carried out by applying surfactants to a pre-cleaned substrate [1–4].

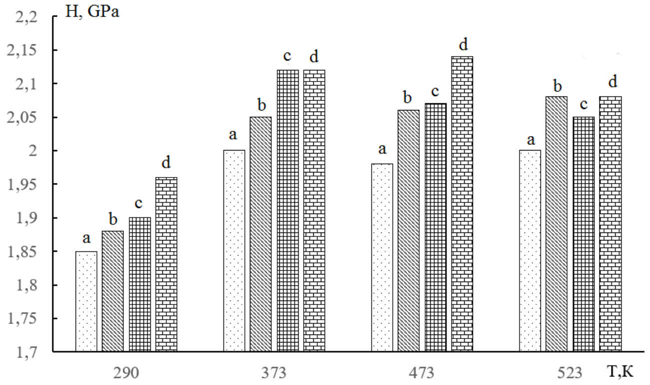
According to modern concepts, the application of surfactants to the surface of a polycrystal leads to a decrease in strength. This strength-reducing effect (meaning the external adsorption effect, when volume diffusion and corrosion phenomena do not occur) is caused by the facilitation of dislocation emergence at the surface of the deformation crystal as a result of a decrease in the surface energy of the solid during surfactant adsorption [4–6]. This effect is widely used in the plastic deformation of metals. However, in a number of cases, when treating crystals with surfactants based on fluorine-containing oligomers, an increase in the microhardness and strength of metals is observed [4, 6, 7].

The aim of the work was to study the strength properties of polycrystals modified with surfactants based on fluorine-containing oligomers, including when exposed to energy factors.

Study methodology and some results

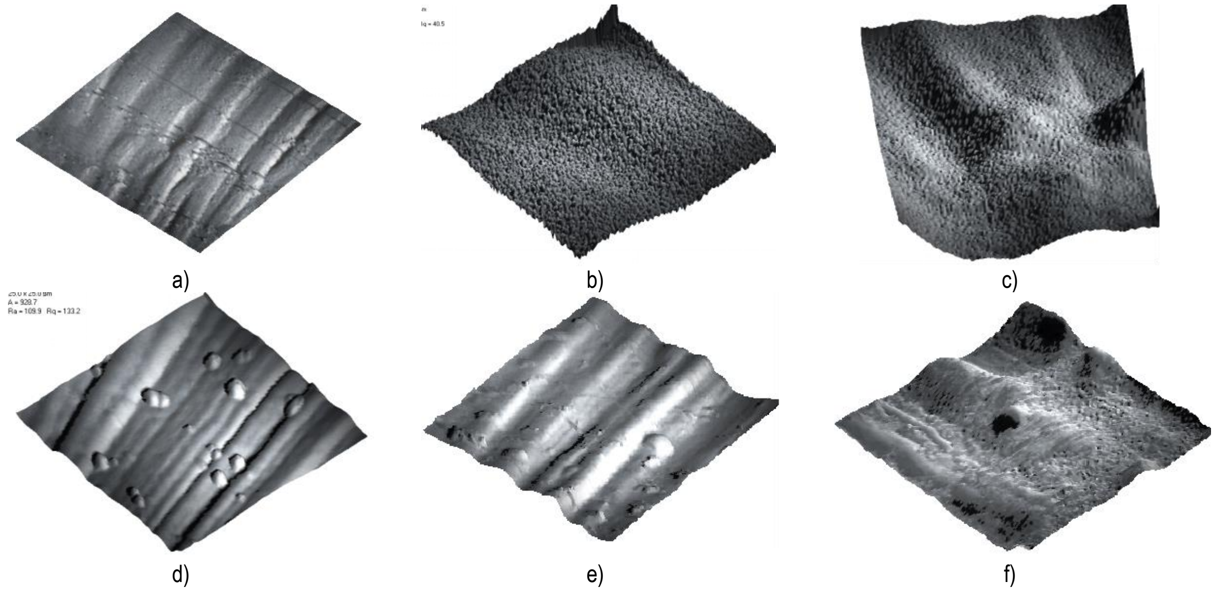
The following metals were used as a substrate: copper (M1); steel 45; aluminum (Al 99); titanium nitride. Fluorine-containing oligomers with the trade name "Foleox" were used as surfactants; their structural formula is R_F – COOH (F1); R_F – CONHR $_1$ (F-AK1); R_F – R_F (F14) (where R_F is a fluorine-containing radical), R_F – CONHR $_2$ (F-AK2), B 1 – water-soluble foleox. The coatings were applied by dipping the substrate in a 1–2 % solution of fluorine-containing oligomer in freon 113, followed by drying in air. Heat treatment of metals with a FSO coating was carried out in air for 1 hour at T = 373 K; 473 K; 573 K. Irradiation was carried out on a URS – 1,0 setup at a voltage of 40 kV and a current of 20 μA on a molybdenum anode (wavelength K $_\alpha$ = 0,7 (A)). The irradiation time was varied from 0 to 60 min. The morphology of the FSO film was examined on an NT 206 atomic force microscope. Microhardness was measured on. The structure of the boundary layers was studied by IR spectroscopy (AFR).

Figure 1 shows the dependence of the microhardness of a steel substrate treated with different grades of fluorine-containing oligomers on the heat treatment mode. The application of fluorine-containing oligomers in all cases leads to an increase in the microhardness values. This can be explained by the process of chemisorption of polar molecules on the surface of the steel substrate, which results in the healing of microdefects in the surface layers of polycrystals and an increase in the free energy of the surface layers of the metal, which hinders the emergence of dislocations on the surface [6]. Chemisorption of these grades of foleoxes on the metal surface is confirmed by the appearance of an absorption band in the IR spectrum in the region of 1610–1690 cm^{-1}, which is identified as the absorption band belonging to the metal salts $(COO)_2$ Me, Me (NH_2), Me (NH_3) [7].



a – steel 45; b – steel 45 + B1; c – steel 45 + FAK-2; d – steel 45 + F14
Figure 1 – Dependence of the microhardness of steel treated with fluorine-containing oligomers on the heat treatment mode

The optical density of the absorption band depends on the grade of foleox used, i. e., on its structure. Heat treatment leads to a further increase in microhardness values. As a result of energy exposure, crystallization processes occur in thin-layer composite coatings (Figure 2), which may explain the effect of increased microhardness values [8–11].



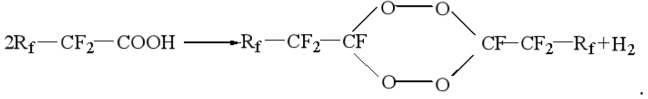
a – original steel substrate; b – steel substrate + Folekos F1; c – steel substrate + Folekos F1, heat treatment at 473 K for 1 hour;
d – steel substrate + Folekos F1, heat treatment at 573 K for 1 hour; e – steel substrate + Folekos F1, X – ray irradiation for 15 minutes;
f – steel substrate + Folekos F1, X – ray irradiation for 60 minutes (scanning area 12 × 12 µm)
Figure 2 – Morphology of oligomer films on metals depending on the energy treatment

Foleoxes F1 and F14 are fluorine-containing compounds containing chemically active groups: COOH (F1) and CF2 = CF (F14). Exposure of FSO to radiation results in noticeable changes in both the ATR and external reflectance spectra, with the difference that the changes in the ATR spectra are more significant. This indicates that, although the process affects the entire volume of the FSO, it is diffusion-controlled and is apparently related to the action of atmospheric oxygen [12–14].

Qualitatively, the spectral changes are expressed in a decrease in the optical density of the absorption bands at 980 and 1280 cm^{-1}, an increase in the optical density at 1340 $cm^{-1,}$ and the appearance of additional peaks in the region of wavenumbers of 1600-1900 cm^{-1}. In the F1 spectra, there is a decrease in the optical density of the 1640 cm^{-1} band with the simultaneous appearance of absorption bands at about 1720 and 1780 cm^{-1}, which can be attributed to C = O bonds in organofluorine compounds.

Irradiation of Foleox F1 on an aluminum substrate leads to the appearance of absorption bands in the ATR and BO spectra in the region of 1700–1800, 1870–1900 cm^{-1}, which can be attributed to peroxide and hydroperoxide groups [15–17].

Transformations of this kind are characteristic of organofluorine acids and can occur according to the scheme



Based on the properties of fluorinated acids, oxidation under the influence of oxygen promotes the formation of free radicals, which leads to the formation of peroxy acids $R_F – COOH$ ® RCOOOH.

Another possible process under these conditions may be decarboxylation with the formation of unsaturated compounds:

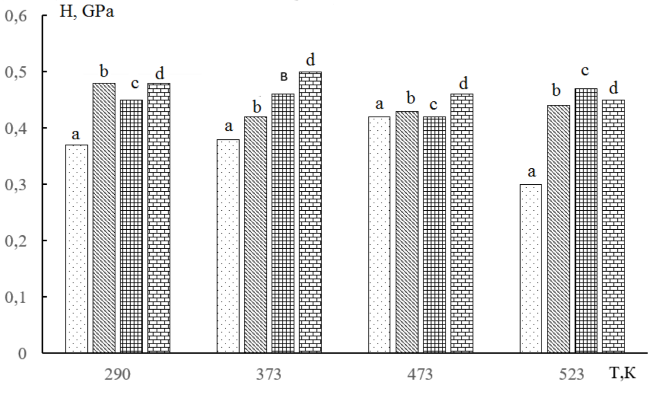
R_F–COOH ® R_f – CF_2H + CO_2
R_f–CF_2H ® R_f – CF +HF
R_f–CF + R_2 – CF ® R_f – CF = CF – R_2.

Unsaturated compounds are capable of reacting with peroxyacids to form cross-linked structures. The appearance of absorption bands at 1700–1800 cm^{-1} in the spectra is consistent with the proposed mechanism.

It is obvious that transformations of this kind must be carried out with the participation of the substrate as a catalyst. It is known that copper and iron are capable of forming carboxyl binuclear complexes with metal-metal bonds, which are catalysts for many transformations. The absorption bands at 1540–1670 cm^{-1} correspond to the absorptions of the carboxyl groups COO – in complex compounds with Al, Cu and Fe.

Irradiation of F1 deposited on a copper substrate leads to a decrease in the intensity of the absorption band at 1660 cm^{-1} with a simultaneous increase in absorption in the region of 1520–1600 cm^{-1} [18, 19].

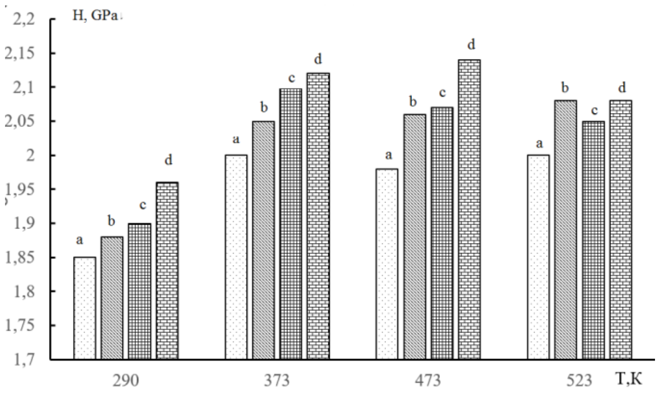
Irradiation of FSO grade F1 film on an iron sample leads to increased unsaturation and, with longer exposure times, to oxidative processes. The orientation of CF and CF2 bonds is perpendicular to the substrate plane, while the C–C bonds are parallel to the substrate. The C–O bonds formed as a result of irradiation are oriented perpendicular to the substrate. With increasing irradiation time, the orientation increases for both F1 and F14 [20].



a – aluminum + FAK-2; b – aluminum + F14; c – aluminum; d – aluminum + B1
Figure 3 – Dependence of the microhardness of aluminum treated with fluorine-containing oligomers on the heat treatment mode

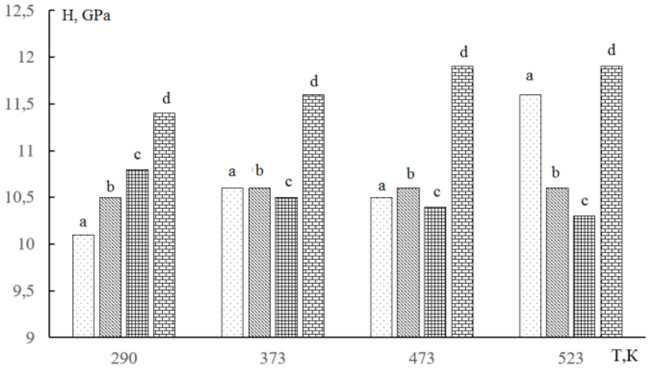
At temperatures above 523 K, ~destructive processes occur in fluorine-containing oligomer films, resulting in film desorption from the metal surface and a consequent reduction in microhardness. Applying fluorine-containing oligomers to an aluminum substrate sometimes leads to a decrease in microhardness (Figure 3).

IR spectroscopy studies of the structure of fluorine-containing oligomer films revealed that chemisorption bonds do not form upon application of fluorine-containing oligomers, resulting in the Rehbinder effect. Heat treatment induces crystallization processes in thin fluorine-containing oligomer films. Figures 4 and 5 show the microhardness curves for copper and titanium nitride substrates treated with various grades of fluorine-containing oligomers. As in the previous cases, an increase in microhardness is observed when chemisorption bonds form between fluorine-containing oligomer molecules and the substrate. If this process does not occur, plasticization of the polycrystal is observed. Heat treatment virtually always leads to an increase in microhardness.



a – copper, b – copper+FAK2; 3 – copper+F1; 4 – copper+B1
Figure 4 – Dependence of the microhardness of copper treated with fluorine-containing oligomers on the heat treatment mode

Another mechanism explaining the increased microhardness of polycrystals may be that oligomer molecules "flow" into the indentation after the indenter is removed from the test material. This results in a decrease in the indentation diagonal and, consequently, an increase in microhardness. Studies have shown that this effect has virtually no effect on microhardness measurements.



a – titanium nitride + fluoroplastic; b – titanium nitride + FAK2;
b – titanium nitride + F14; g – titanium nitride + FAK1, titanium nitride + F1
Figure 5 – Dependence of the microhardness of titanium nitride treated with fluorine-containing oligomers on the heat treatment mode

According to classical concepts, the application of surfactants to metal surfaces should lead to the implementation of the Rehbinder effect [9].

Typically, two types of the Rehbinder effect are distinguished: 1) external and 2) internal. The external Rehbinder effect consists in the fact that upon adsorption of surface-active substances (surfactants), the free energy of solids decreases. This reduces the resistance of the surface layer of the solid to plastic deformation, facilitates plastic flow in grains and the emergence of dislocations to the surface [9]. The upper layer of the metal may have lower microhardness than the underlying layers saturated with dislocations. The internal Rehbinder effect (adsorption-wedging) is realized during the adsorption of molecules on the surfaces of cracks occurring in the surface layer of the solid. The active centers of the molecules reach an area whose size is less than two sizes of the molecules, the latter, attracted by the crack walls and experiencing the pressure of neighboring molecules, tend to wedge it. This phenomenon contributes to the destruction of the modified surface layer. However, in the works [10–13] it was shown that the application of surfactants such as fluorine-containing oligomers to metal substrates in a number of cases leads to strengthening of the surface layers of the material and the Rebinder effect does not appear.

The application of fluorine-containing oligomers to metals results in the formation of nanophase composite materials that exhibit an electret state, as evidenced by the presence of conduction current values ranging from |1–5| pA under normal experimental conditions. It was found that increasing the thickness of the fluorine-containing oligomer coating leads to a decrease in conduction current values, which is explained by the shielding effect of the fluorine-containing oligomer coating on the charge mosaic of solid surfaces. However, exceeding the thickness of the fluorine-containing coating on a metal surface above 1,0–1,2 µm reduces the shielding effect due to disordering in the boundary layers of the coating. The charge activity of the metal substrate significantly influences the activity of the resulting fluorine-containing coating. The presence of picoampere currents in coatings formed from non-polar fluorine-containing oligomers was established, with these currents exceeding those generated in coatings formed from polar oligomers. This effect can be explained by the fact that chemisorption of polar fluorine-containing oligomers most likely occurs primarily at the active (charge) centers of the metal surface. Changes in thermally stimulated currents in coatings formed from non-polar oligomers on a metal substrate are virtually independent of the coating thickness when the metal surface is treated with fluorine-containing oligomers more than twice. Coatings formed from a combination of polar and non-polar oligomers produce protective layers that combine high chemical adsorption activity provided by the polar oligomer with a surface layer formed from a non-polar oligomer with low shear strength. These coatings exhibit a high shielding effect on the charge surface of the solid.

Conclusions

Fluorinated oligomers strengthen the surface layers of the substrate if chemical (chemisorption) bonds, such as salts of higher acids, are formed between the film. A fluorine-containing oligomer and a metal (substrate) are combined. If chemisorption interactions between the oligomer molecules and the substrate do not occur, plasticization of the surface layers of the polycrystals occurs. Energy treatment intensifies crystal formation processes in thin films, resulting in increased microhardness of the thin-film composite coating. These coatings are recommended for friction units of construction equipment operating in harsh conditions.

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Material received 26.09.2025, approved 16.10.2025, accepted for publication 30.10.2025