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ANALYSIS OF THE TRIBOLOGICAL PROPERTIES OF Cu-ATiO$_2$ COMPOSITE COATINGS APPLIED BY THE COLD SPRAY METHOD

ANALIZA WŁAŚCIWOŚCI TRIBOLOGICZNYCH POWŁOK KOMPOZYTOWYCH Cu-ATiO$_2$ NANOSZONYCH METODĄ COLD SPRAY

Key words: composite coatings, LPCS, friction, abrasive wear.

Abstract: The properties of copper have been known and used for a very long time, and research has also been carried out for a long time to expand the applications of this material. One of the methods increasing the bactericidal and bacteriostatic effect of copper is modification by means of the TiO$_2$ phase. The research was conducted in order to determine the impact of modification of copper coatings with TiO$_2$ titanium dioxide on their tribological properties. The paper presents the results of studies on tribological wear of composite coatings applied on steel using the method of low-pressure cold gas spraying (LPCS). The tests of resistance to abrasive wear were carried out in a ball-disc combination in reciprocating motion. The analysis of the resistance to abrasive wear of the tested coatings included the determination of the impact of the pressure force on the intensity of wear and the kinetic friction coefficient of the tested friction pairs. It was found that the samples covered only with copper coatings were characterized by a higher value of friction coefficient in relation to the substrate made of AISI 316l steel. The modification of copper with the submicron particles TiO$_2$ fraction does not increase the value of friction coefficient. The value of this parameter is maintained at a similar level regardless of the applied counterspecimen.

Słowa kluczowe: powłoki kompozytowe, LPCS, tarcie, zużycie ścierne.

Streszczenie: Właściwości miedzi są znane i wykorzystywane od bardzo dawna, od dawna również są prowadzone badania nad zwiększeniem możliwości zastosowania tego materiału. Jedną z metod zwiększających działanie bakteriobójcze i bakteriostatyczne miedzi jest modyfikacja fazą TiO$_2$. Badania przeprowadzono w celu określenia wpływu modyfikacji powłok miedzianych ditlenkiem tytanu TiO$_2$ na ich właściwości tribologiczne. W pracy zaprezentowano wyniki badań zużycia tribologicznego powłok kompozytowych nanoszonych na stal metodą niskociśnieniowego natrysku zimnym gazem (LPCS). Badania odporności na zużycie ścierne wykonano w skojarzeniu kula–tarcza, w ruchu posuwisto-zwrotnym. Analiza odporności na zużycie ścierne badanych powłok obejmowała określenie wpływu siły nacisku na intensywność zużywania i współczynnik tarcia kinetycznego badanych par trących. Stwierdzono, że próbki pokryte wyłącznie powłoką miedzianą charakteryzują się wyższą wartością współczynnika tarcia, w stosunku do podłoża ze stali AISI 316l. Modyfikacja miedzi frakcją submikronowych cząstek TiO$_2$ nie powoduje zwiększenia wartości współczynnika tarcia. Wartość tego parametru zostaje zachowana na podobnym poziomie niezależnie od zastosowanego obciążenia przeciwpróbki.

INTRODUCTION

The properties of copper and its alloys have been used for centuries in many areas of life and science. In addition to its aesthetic qualities, copper has a number of physicochemical properties that allow ever new applications. New technologies and scientific discoveries open almost unlimited application possibilities **[L. 1–4]**.

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The antimicrobial activity of copper and copper-based alloys is also well documented, and copper has recently been registered in the USA with the Environmental Protection Agency as the first solid antimicrobial material **[L. 1–4]**. As a result of clinical studies, it was found that copper can be used on touchable surfaces such as door handles, bathroom fittings or bed rails to reduce nosocomial infections **[L. 5]**. At a time when there is a global epidemic, caused by a huge number of cases and deaths due to COVID-19, this issue is particularly relevant. The global situation makes it necessary to extend these issues also to coatings on elements for everyday use. At present, the elements on which micro-organisms persist are those with which we are in contact in public places (shops, parks, entertainment centres); they constitute the greatest threat and on them the COVID-19 virus takes the greatest toll.

Literature reports that copper-based composite coatings offer interesting results and possibilities by, e.g., increasing microbiological activity **[L. 6–16]**. To diversify and improve the microbiological activity of copper, both TiO_2 and Ag are used, either in the form of an interlayer or modifier. The addition of active ceramics is intended to increase the bactericidal activity of the surface, and the addition of TiO_2 phase also uses photocatalytic activity **[L. 17–22]**.

The method of low-pressure cold gas spraying (LPCS) allows to apply both soft metals (Cu, Al, Zn, Sn) using the mechanism of plastic deformation of powder particles, as well as modification of the base powder with ceramic particles (SiC, Al_2O_3, TiO_2) **[L. 20, 21, 23]**. Usually, the presence of a hard ceramic fraction causes strengthening of the coating and the reduction of porosity of the soft metallic matrix. It is different in case of the amorphous form of $aTiO_2$, which, due to its structure, does not cause a stronger degree of crushing of metallic matrix **[L. 24–26]**. Low temperature (400°C) during the LPCS process does not cause phase changes in $aTiO_2$, leaving the ceramic phase $aTiO_2$ in amorphous form. Scientific research **[L. 6, 7, 18]** confirms that this form of $aTiO_2$ also shows biological and physicochemical activity, causing changes in the properties of the base material, e.g., Co and Cu. Our previous research **[L. 12]** has shown that using LPCS methods makes it possible to produce durable Cu-TiO_2 composite coatings. Additionally, on the basis of our own research, it was found that $aTiO_2$ modification results in the shift of corrosion potentials towards more positive values, which means an increase in corrosion resistance of Cu-$aTiO_2$ composite coatings. However, there are no reports in the literature about testing mechanical properties of this type of composite coatings. Therefore, the study presented in this paper was aimed at determining the impact of modification of copper coatings with a fraction of amorphous titanium dioxide on tribological properties of coatings applied by the Cold Spray method. Bioactive coatings for applications on public utility surfaces (handles, handrails, and others) should be durable, have appropriate thicknesses, and be resistant to frequent touching and friction. Therefore, relevant tests presented in this publication have been undertaken.

MATERIALS AND MEASURING METHODS

Tribological studies were carried out on copper coatings and copper coatings modified with wt10% of amorphous titanium dioxide. Commercially available copper powder of dendritic morphology and fraction size from 10 µm to 50 µm was used. Amorphous submicron titanium dioxide $aTiO_2$, produced using sol-gel technology, with submicron granulation in the range of 400–600 nm, which forms agglomerates of 10–70 µm, was used for the modification **[L. 25]**. The mass fraction of titanium dioxide was selected on the basis of preliminary tests of coating microstructure. Powders were mixed with the use of laboratory mill for t = 60 min. Copper powders and Cu-$aTiO_2$ composite powders were applied on an AISI 316L austenitic steel substrate, 10x70 mm in size and 1 mm thick. Before spraying, the substrate was blasted at a pressure of p = 0.6 MPa and using electrocorundum of 0.8 mm granulation. Low-pressure cold gas spraying (LPCS) was used to apply the coatings at the technological parameters presented in **Table 1** (determined by means of an experiment). The porosity was determined using a graphical semi-quantitative analysis using the ImageJ software. Five images with a magnification of 2500x were selected, and the average value from the measurements was taken as the result. The coating thickness after application was 600 µm for the copper coating and 200 µm for the composite coating (due to different particle sizes of Cu and $aTiO_2$ the deposition of coatings is more difficult). An analysis of the surface topography of coatings after the mechanical polishing process (using 1 and 6 µm polishing slurries) was carried out before and after tribological tests in order to evaluate changes in coatings.

Table 1. Technological parameters of coating application using the LPCS method

Tabela 1. Parametry technologiczne nanoszenia powłok metodą LPCS

Powder	Gas heating temperature	Gas pressure	Sliding speed
Cu, Cu-$aTiO_2$	400°C	0.9 MPa	10 mm/s

Friction tests were carried out on a ball-on-plate test stand which enables one to carry out sliding friction tests in a reciprocating motion **[L. 27]**. A silicon carbide SiC ball with a diameter d = 3.969 mm was used for testing. The counterspecimen was a steel plate made of AISI 316L steel with an applied coating.

During the tests, the ball was pressed against the counterspecimen by F_n with three different values: $F_n = 5$ N, $F_n = 10$ N and $F_n = 15$ N. A steel plate with an applied coating was fixed on a movable carriage. Three series of measurements were carried out in the test, and, in each of them, the plate mounted on a moving trolley performed a series of 200 movement cycles. The movement cycle consisted of two movements in both directions, achieving a maximum relative speed $v_{smax} = 5$ mm/s with a stroke of $S = 10$ mm. The motion time in each direction was $t = 0.4$ s. During the measurements, the friction force values were recorded at a frequency of $f = 100$ Hz, and the measurement limit error determined based on the sensor and amplifier class was $\Delta gr = 0.05$ N. The tests were conducted under technically dry friction conditions. The obtained measurement results were subject to statistical calculations.

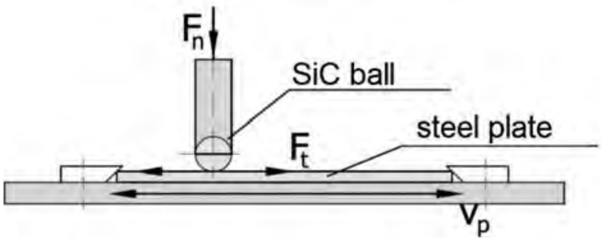

Fig. 1. Diagram of the test stand for alternating motion using a ball-on-plate combination [L. 28]

Rys. 1. Schemat stanowiska do badań w ruchu przemiennym z wykorzystaniem skojarzenia typu ball-on-plate **[L. 28]**

Five hardness measurements were carried out for each coating material, in accordance with the PN-EN ISO 6507-1:2007 standard, using the Vickers method and a Leco LM-248AT hardness meter. The measurements were carried out at a load of $m = 1$ kG (9.81 N). Analyses of the coating surface after friction with the use of scanning electron microscopy methods were carried out with the use of a SEM Phenom G2 microscope, using a BSE detector.

RESULTS AND DISCUSSION

Coatings obtained in the LPCS process show a compact and homogeneous microstructure in terms of morphology but are characterized by different degrees of plastic deformation. Thus, there is a different degree of deformation and strengthening of individual coating particles. The different structure has a significant impact on the hardness of coatings (**Table 2**). An averaged five measurements of hardness of coatings, determined in the cross-section, for a copper coating, show a hardness of 86 HV1. Modification with the amorphous ceramic phase TiO_2 causes a decrease in hardness to 63 HV1.

Table 2. Averaged measurements of hardness of applied coatings

Tabela 2. Uśrednione pomiary twardości naniesionych powłok (n = 5)

Coating	HV1	Deviation
Cu	86	8.7
Cu-aTiO_2	63	6.5

The analysis of the surface of samples with Cu coatings showed a small degree of porosity. A lower density of Cu coatings is visible in areas that were not matched entirely in highly developed dendritic copper powder particles (**Figs 2a** and **b)**. In Cu-aTiO_2 composite coatings, potential porosity areas are filled with the ceramic phase aTiO_2. The aTiO_2 inclusions are visible in the form of black inclusions, clearly accumulated within the inter-dendritic spaces of the copper powder (**Fig. 3a**). In case of composite coatings, porosity is visible within the presence of aTiO_2 particles themselves (**Fig. 3b**). It is connected with amorphous structure and lack of plastic deformation of aTiO_2 particles during LPCS application. Due to high ductility, dendritic copper particles, as a result of collision with the substrate, are strongly deformed and fill the space between the particles tightly. Cu coatings show a porosity of 1–2 %. In the case of composite coatings, submicron aTiO_2 particles are unevenly distributed in the inter-dendritic spaces of copper powder. In composite coatings, uneven filling of spaces between metallic matrix particles is visible, i.e. they show higher porosity in relation to copper coatings at the level of 3–4%.

The average value of the kinetic friction coefficient in the combination SiC/Cu coating and the lowest load $F_n = 5$ N was $\mu = 0.33$. Further studies have shown that the addition of 10% aTiO_2, causes this coefficient to remain at a similar level (**Fig. 4**). In the case of a copper coating, a decrease in this coefficient was observed with an increase in the loading force from $\mu = 0.35$ to $\mu = 0.28$. The error bar values allow us to conclude that the most stable measurement conditions can be obtained for the tested pair with the lowest loading force $F_n = 5$ N. With higher loads, i.e. $F_n = 10$ N and $F_n = 15$ N, the spread of the results even reaches approx. 30% of the values obtained.

For the analysis of wear, a method of measuring the width of the friction traces was used to supplement the qualitative assessment of wear **[L. 28–35]**. The analysis of the friction traces produced by the friction processes clearly indicates that the highest wear is observed at the highest load applied, i.e. $Fn = 15$ N (**Fig. 5**). In this range, the width of the friction trace takes the same values, at $x = 400$ µm. This shows that a 10% share of the modifying phase under the specified loads does not affect the tribological properties. For smaller load values, the reduced width of the friction traces is obtained for the modifying sample and the load $F_n = 5$ N, the reduction of

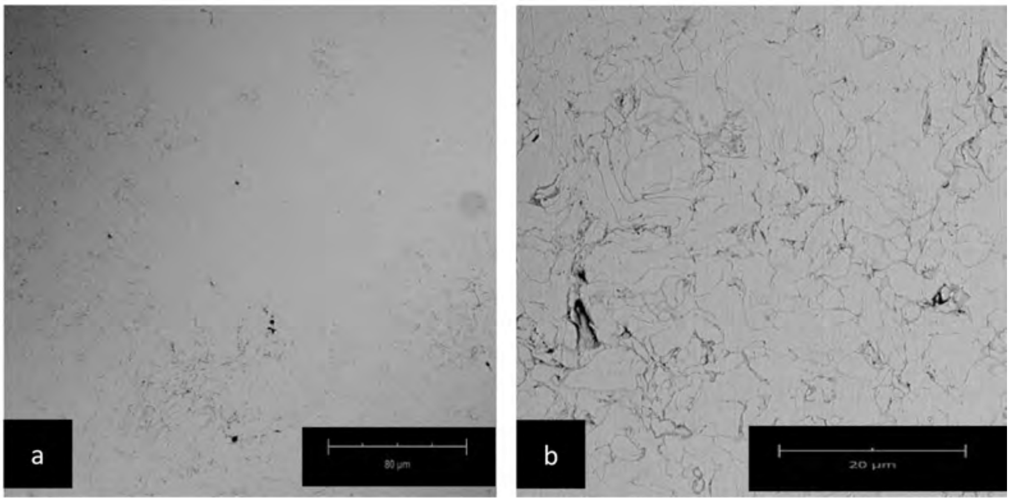

Fig. 2. Cu coating surface, before tribological tests (SEM): a) morphology of the Cu coating, b) enlarged fragment from the area a) with boundaries between the grains of the Cu powder

Rys. 2. Powierzchnia powłoki miedzianej Cu przed testami tribologicznymi (SEM): a) morfologia powłoki Cu z niewielkim stopniem porowatości, b) powiększony fragment z obszaru a) z widocznymi granicami pomiędzy ziarnami proszku Cu

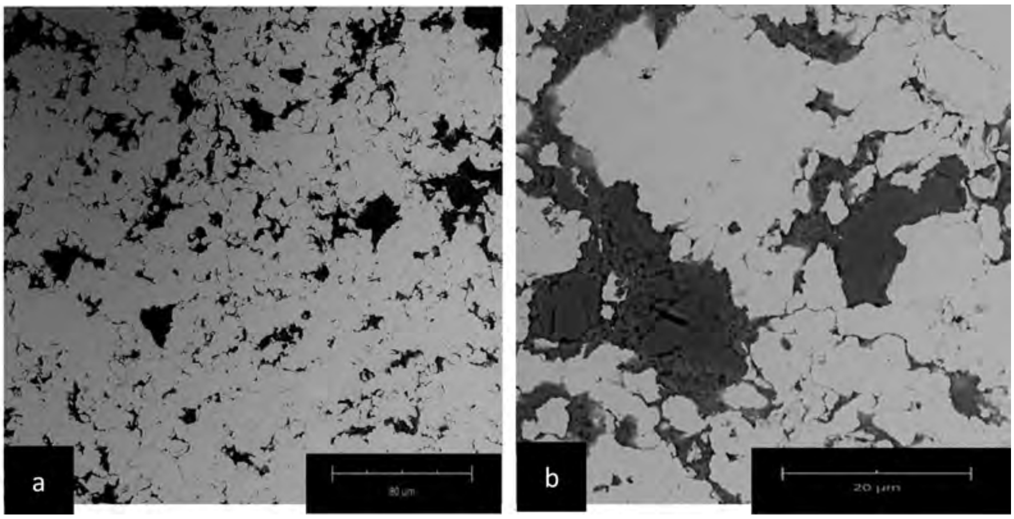

Fig. 3. Surface of Cu-aTiO$_2$ composite coatings, before tribological tests (SEM): a) morphology of the Cu-aTiO$_2$ coating, b) enlarged fragment from area a)

Rys. 3. Powierzchnia kompozytowych powłok Cu-aTiO$_2$ przed testami tribologicznymi (SEM): a) morfologia powłoki Cu (jasny obszar) – aTiO$_2$ (ciemny obszar), b) powiększony fragment z obszaru a)

the friction trace width from $x = 280$ µm to $x = 240$ µm. For both loads $F_n = 5$ N and $F_n = 10$ N in the case of the copper coating, no changes in the width of the friction trace were observed; they remain at a similar level of $x = 280$ µm. A reduction in the width of the friction trace at lower loads indicates greater resistance to the abrasion of composite coatings. The wear of the silicon ball at the given parameters of the experiment was not measurable.

An analysis of the topography of the coatings surface after tribological tests was also carried out using scanning electron microscopy methods. The tests showed that, as a result of contact between the coatings with the ball, the coatings become worn evenly. The nature of wear was determined as plastic, and in no case were any brittle splinters of coatings observed. Both copper coatings Cu (**Fig. 6**) and composite coatings Cu-aTiO$_2$ (**Fig. 7**) show a similar mechanism of wear without the occurrence of areas of brittle cracks, and the addition of the modification phase does not change this mechanism. This is also confirmed by the friction coefficient values recorded for both types of coatings. Modification with amorphous aTiO$_2$ fraction, as opposed to crystalline particles, does not cause any cracking in the coating **[L. 19, 28, 32–35]**. The use of amorphous titanium dioxide powder, due to its submicron granulation and additionally an amorphous form, does not lead to brittle cracking.

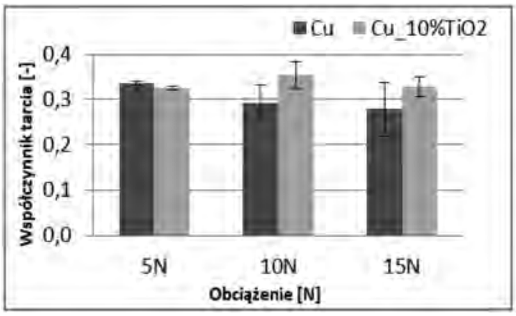

Fig. 4. Averaged friction coefficient values for copper coatings and Cu-aTiO$_2$ composite coatings for various values of loading force

Rys. 4. Uśrednione wartości współczynnika tarcia kinetycznego w badanych skojarzeniach dla różnych wartości siły obciążającej

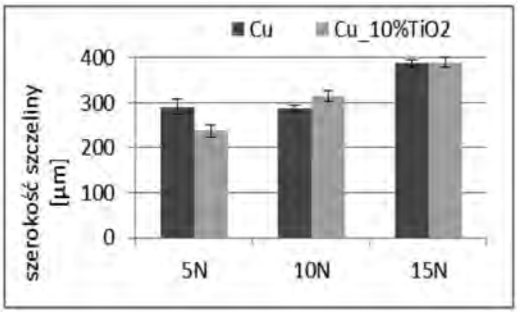

Fig. 5. Average values of the friction path width after friction tests, for Cu coatings and Cu-aTiO$_2$ composite coatings, for different values of loading force

Rys. 5. Uśrednione wartości szerokości śladu tarcia po próbach tarcia, dla powłok miedzianych oraz kompozytowych powłok Cu-aTiO$_2$ dla różnych wartości siły obciążającej

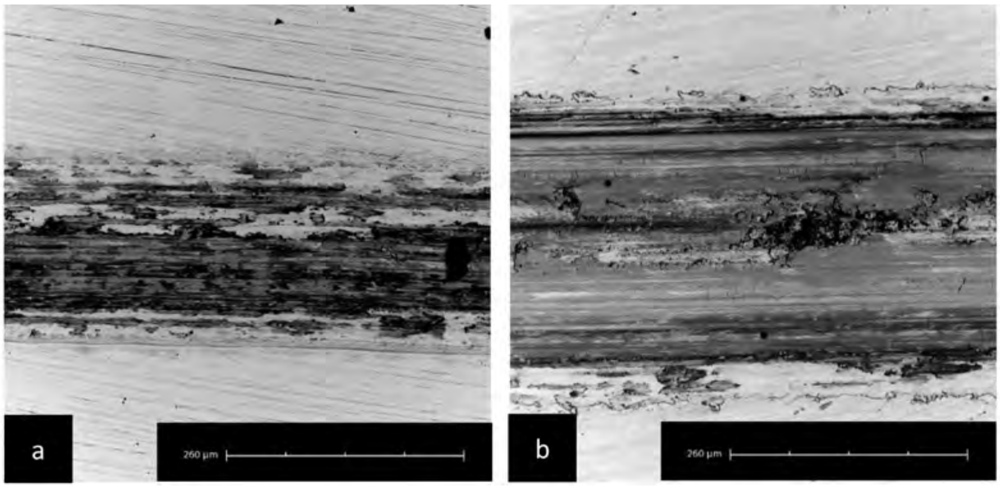

Fig. 6. Examples of wear and tear formed after tribological tests carried out on a Cu coating under load (SEM): a) F_n = 5 N, b) F_n = 15 N

Rys. 6. Przykładowe ślady zużycia powstałe po badaniach tribologicznych przeprowadzonych na powłoce miedzianej Cu przy obciążeniu (SEM): a) F_n = 5 N, b) F_n = 15 N

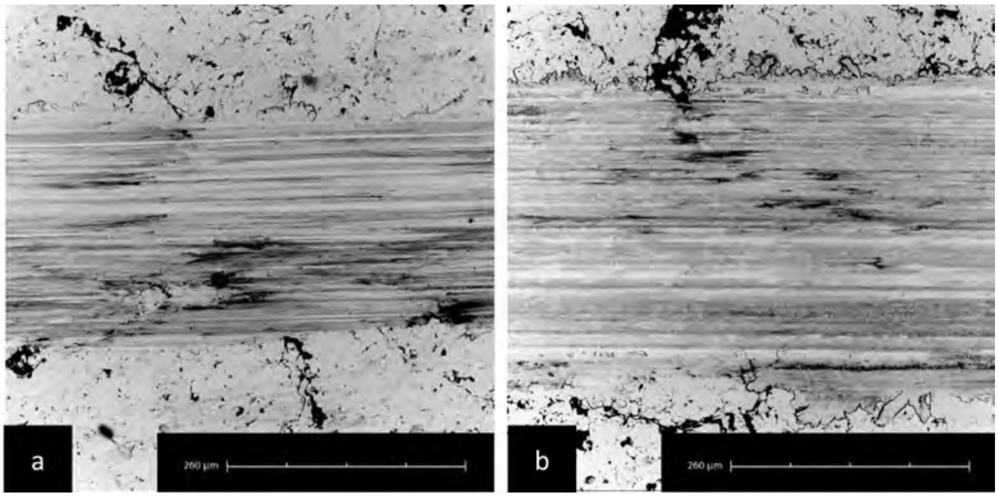

Fig. 7. Examples of wear and tear formed after tribological tests carried out on composite Cu-aTiO$_2$ coating under load (SEM): a) F_n = 5 N, b) F_n = 15 N

Rys. 7. Przykładowa ślady zużycia powstała po badaniach tribologicznych przeprowadzonych na kompozytowej powłoce Cu-aTiO$_2$ przy obciążeniu (SEM): a) F_n = 5 N, b) F_n = 15 N

CONCLUSIONS

The tribological and instrumental studies lead to the following conclusions:

- The addition of a ceramic phase in the form of 10% by weight of amorphous aTiO_2 reduces the hardness of coatings. The recorded decrease of hardness value is 86 HV1 for copper coating and 63 HV1 for Cu-aTiO_2 composite coating.
- The change of load (5, 10, 15 N) does not affect the friction coefficient in combination with the composite coating.
- For the Cu-aTiO_2 composite coating, the value of the friction coefficient is maintained at a similar level regardless of the load value, taking into account the error bars obtained.
- Observation of the wear marks on the coatings as a result of the friction process showed that both copper coatings and Cu-aTiO_2 composite coatings show a high degree of plasticity. Observation of images recorded with the use of scanning electron microscopy methods showed that there are no brittle changes in coatings.
- The research shows that modification of the copper coating with a 10% share of titanium dioxide does not trigger the deterioration of tribological properties of coatings. This allows the extension of the potential application of composite coatings to biologically active coatings used on elements of public use (handles, handrails, and others).

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