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Research article

Working Out the Processes of Deposition "Metal-Metal" Multi-Layer Coatings (Cu-Mo, Cu-MoN, Cu-C) and Studying the Tribological Characteristics of Friction Pairs

Olexiy Sagalovich[a,*], Viktor Popov[a], Vladislav Sagalovich[a], Stanislav Dudnik[a], Oleksandr Olijnyk[b]

[a]*JSC "FED", Kharkiv, Ukraine,*
[b]*Machine building plant Malishev, Kharkiv, Ukraine.*

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* Corresponding author:

Olexiy Sagalovich
E-mail: iht@kharkov.ua

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A B S T R A C T

As part of the program to search for new materials with high characteristics according to Avinit vacuum-plasma technologies, based on the complex use of coating methods (CVD + PVD), stimulated by non-equilibrium low-temperature plasma, the technological parameters of applying tightly bonded high-quality multilayer and nano-layer "metal-metal coatings" were worked out:

- *metal PVD coatings of Mo, Cu;*
- *multi-layer PVD coatings based on Cu-Mo-N;*
- *multi-layer PVD coatings on the basis of (Cu-C) (with different carbon content).*

Studies of samples with coatings were performed (micro-grinding, coating hardness, determination of surface geometry after coating). The thickness of multilayer coatings is 1...2 microns. with coating hardness of 250-400 kg/mm². The developed regimes ensure high adhesion and preservation of the hardness of steel DIN 1.2379 within the specified limits. Distortions of the geometry and roughness of the coated surfaces, compared to the condition before the coating, were not detected.

Tribological tests of coated samples were carried out. All tested coatings were completely worn out when rubbing steel DIN 1.2379 at very low loads (max up to 300N, then the load did not increase). All coatings had a high coefficient of friction Kfr ≥ 0.15. The use of such coatings in tribocombinations with steel in the form of independent wear-resistant coatings is impractical. They can be considered as soft layers in the development of new structures of anti-friction wear-resistant coatings to increase the performance of friction pairs in the "coating-steel" and "coating-coating" tribosystems.

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1. INTRODUCTION

It is usually considered that high hardness is the main requirement for the material that is laid in the design of the device, if its surface will work for wear. From this point of view, ceramic coatings are the best choice for improving the wear resistance of structural materials, as they have high hardness and at the same time high chemical inertness.

In many applications other than tool hardening, where different wear mechanisms are often in competition, ultrahard ceramic coatings are not always the ideal solution. This applies to strengthening cases of relatively soft structural materials with a low modulus value, for which superhard and hard ceramic coatings are fundamentally incompatible with the substrate material. Metal-metal nanocomposite coatings with improved mechanical properties that combine high hardness with good plasticity appear to be more promising in some cases. Such coatings are able to withstand larger deformations in the elastic region, without reaching a critical level of stresses that lead to the formation of cracks in the coating, its local destruction, and discoloration. More elastic coatings are able in the "coating-substrate" combination to provide the ability to plastically deform the substrate as well, which is more important than just having high hardness in most cases of contact interaction of friction surfaces.

The most objective information about the relative wear resistance of different materials is given by the values of hardness H and modulus of elasticity E. The anti-seize properties of materials working in contact with each other largely depend on these characteristics.

Generally, hardness and Young's modulus are somewhat correlated. Knowing their values, it is possible to estimate the resistance of the coating to plastic deformation, which is greater, the higher the H3/E2 ratio. Therefore, along with coatings based on solid compounds, which have long occupied a dominant position in the development of wear-resistant and strengthening coatings, intensive research has recently been conducted on coatings that contain metal or compounds with low hardness, the so-called "metal-metal nanocomposite coatings".

Such coatings are able to withstand large deformations in the elastic region, without reaching a critical level of stresses that lead to the appearance of cracks in the coating, its local destruction, and crumbling. More plastic coatings are able in the "coating-substrate" combination to ensure the possibility of plastic deformation of the substrate as well, which is more important than just having high hardness in most cases of contact interaction of friction surfaces.

An example of such coatings can be TiN-Ti, TiN-Cu, AlN-Cu, Mo-Cu, MoN-Cu, MoN-MoS2 and others [1-5].

Their properties are largely determined not only by the ratio of elements in the coating and the phases included in their composition, but also by structural characteristics. This applies to both single-layer coatings and multi-layer ones, especially the latter. This is most evident in the formation of nanostructured coatings. The main requirements for the selection of materials for nanocomposite coatings and the formation of their structure in order to achieve high values of microhardness are considered in a number of works [6-8].

An example of experimental confirmation of obtaining ultra-hard nanocoatings can be the coating of the Ti-Si-N system of the TiN-Si3N4 type, which can be classified as "classic".

Numerous experimental results testify to the complex and ambiguous nature of the dependence of properties on the composition, structure, and conditions of obtaining coatings, and therefore, today, there is an accumulation of data on the properties of such coatings, taking into account the great diversity both in composition, conditions, and methods of their formation.

In the selection of tribocouple materials to prevent seizure or reduce damage to an acceptable level, along with the concept of "hard and superhard coatings", which are nanolayered or nanocomposite structures that have very high hardness, various methods based on the concept of "soft coatings" are used:

- choose a combination of materials for friction pairs with a minimal tendency to seize;
- the friction surface is coated with soft metals (In, Cd, Sn, Ag, Cu, brass, etc.);
- introduce soft components (Pb, Sn) into antifriction alloys;
- use materials performing solid lubrication functions (graphite, molybdenum disulfide, etc.);

- in tribocombinations, materials are used that have a low adhesion capacity relative to the material of the counterbody (carbon graphite antifriction materials, oxide ceramics);
- Me-DLC coatings, which are intensively developing, are of particular interest [9].

When micro-designing multilayer coatings, it is advisable to create soft layers before applying hard coatings. The effectiveness of softer coatings is due to the fact that the soft layer increases the adhesion of the coating to the substrate, ensures the presence of a large positive gradient of mechanical properties in the coating, which is a good prerequisite for normal operation in friction conditions. Such layers prevent the propagation of cracks that occur during operation. Chromium, molybdenum, α -Ti are usually used as soft layers.

2. OBTAINING SAMPLES

The development of the processes of application of functional multilayer composite coatings was carried out on the automated vacuum-plasma cluster Avinit according to the specified program using single-component cathodes in the medium of reaction gas (nitrogen) and without it [10-12].

Methods of obtaining coatings, studying their metallographic and tribological properties are described in detail in works [10-12].

The following materials and technologies were selected for development:

- metal PVD coatings of Mo, Sg;
- multi-layer PVD coatings based on Cu-Mo-N;
- multi-layer PVD coatings based on (Cu-C).

The technological parameters of obtaining of strongly bonded high-quality metal PVD coatings Mo, Cu, multi-layer and nano-layer PVD coatings of various compositions (Cu-Mo, Cu-Mo-N) and coatings (Cu-C) (with different carbon content) were worked out, as well as multi- and nanolayer compositions based on them on samples made of X12F1 steel with a hardness of 56... 61HRC (nitrided and cemented).
Two technological schemes were used to deposit multilayer coatings of the "metal-metal" type [13]:

a) double-cathode scheme with simultaneous operation of two sputtering sources, which are placed facing each other, in the environment of residual gases with the rotation of the sample around its axis;
b) double-cathode scheme during pulsed operation of two sputtering sources, which are placed facing each other, in the environment of residual gases with the sample rotating around its axis.

2.1 Metal PVD coatings and multi-layer coatings based on Cu-Mo-N

Avinit A/P210 (Mo) and Avinit A/P 310 (Cu) metal coatings were deposited according to the proven modes for metallographic and tribological tests (according to the cube-roller scheme).

Samples with coatings were studied (micropolishing, coating hardness, determination of surface geometry after coating). There are no distortions of the geometry and roughness of the coated surfaces compared to the condition before the coating. The thickness of multilayer coatings is 1...2 microns. The measured hardness values of the coatings were 250 - 400 kH/mm^2 for the tested samples.

The developed regimes ensure high adhesion and preservation of the hardness of steel DIN 1.2379 within the specified limits. Metallographic studies of samples with coatings confirmed the integrity and uniformity of the thickness of the coatings on the entire surface of the samples.

The technological parameters of application of firmly bonded high-quality coatings were optimized, and the modes of application of PVD coatings of different composition (Cu-Mo, Cu-Mo-N) and multi- and nano-layer compositions based on them were worked out.

The developed regimes ensure high adhesion and preservation of the hardness of steel DIN 1.2379 within the specified limits. Metallographic studies of samples with coatings confirmed the integrity and uniformity of the thickness of the coatings on the entire surface of the samples.

A study of samples with coatings was carried out (micro-grinding, coating hardness, determination of surface geometry after coating). Distortions of the geometry and roughness of the coated surfaces, compared to the condition before the coating, were not detected.

The component composition and types of coatings are indicated in the table 1.

Table 1. Samples are coatings applied to the main sample (cube) (steel DIN 1.2379, 56...61HRC)

	Coating	Basic sample		Tested pairs of friction/max load, kN
Samples without coating				
1		023		023 – 6_{10} (nitrided.) /1,4 023 – 6_{11} (nitrided.) /0,8
2		1 2		1 – 7_4 (cemented.) /1,2 1 - 6_7 (nitrided.) /1,4
Multi-layer coatings based on Cu-(Mo-N)				
3	M04/1-1 M04/1-3	031 033	Single layer coating (Cu-Mo-N) Avinit C/P 420	031 - 7_5 (cemented.)/1,4
4	M04/2-1 M04/2-2 M04/2-3 M04/2-4	036 037 038 039	Nanolayered coating Cu-(Mo-N) The ratio of layer thicknesses 1:1 Avinit C/P 420	040 – 7_6 (cemented.) /1,4 040 – 7_2 (cemented.) /1,4
5	M04/3-1 M04/3-2	042 043	Single layer coating (Cu-Mo) Avinit A/P 500	042– 6_8 (nitrided.) /1,4 043 – 7_{12} (cemented.)/1,4
6		021 022 047 048	Nanolayered coating Cu-Mo The ratio of layer thicknesses – 1:2 Avinit A/P 520	048 – 7_3 (cemented.) /1,4
7		19 20 21	Microlayered coating Cu-Mo The ratio of layer thicknesses – 2:1 Avinit A/P 510	19 – 6_9 (nitrided.) /1,0
8		23 24 25	Microlayered coating Cu-(Mo-N) The ratio of layer thicknesses – 2:1 Avinit C420	23 – 6_{12} (nitrided.) /1,6 23 – 7_7 (cemented.) /1,2

2.2 Metal PVD coatings and multi-layer coatings based on Cu-Mo-N

The technological parameters of deposition of firmly bonded high-quality multilayer and nano-layer coatings (Cu - C) (with different carbon content) with a thickness of 1...2 microns on counterbodies of rollers (nitrided and cemented steel) were worked out.

The developed regimes ensure high adhesion and preservation of the hardness of steel DIN 1.2379 within the specified limits. Metallographic studies of samples with coatings confirmed the integrity and uniformity of the thickness of the coatings on the entire surface of the samples.

Received samples with multi-layer and nano-layer coatings (Cu – C) on counterbodies (rollers) for tribological tests.

Multi-layer and nano-solder coatings (Cu-C) of three types (with different carbon content) were applied to the counterbodies (rollers) for tribological tests according to the developed regimes:

-Cu-C(l); -Cu-C(II); - Cu - C (lll).

A study of samples with coatings was carried out (micro-grinding, coating hardness, determination of surface geometry after coating).

The thickness of multilayer coatings is 1...2 microns. The measured values of the hardness of the coatings on the rollers were 250-400 kg/mm2 for the tested samples.

3. CARRYING OUT TRIBOLOGICAL TESTS OF COATED SAMPLES

3.1 Metallic PVD coatings Mo, Cu and multi-layer coatings based on Cu-Mo-N

Tribological tests of samples with "soft" coatings (metal PVD coatings Mo, Cu and multi-layer coatings based on Cu-Mo, Cu-Mo-N.

The test results are given in the table 2. The absolute loads, kN, coefficients of friction, the maximum achieved loads to burr on the samples after tests in the investigated pairs are given.

Studies of samples with coatings (micro-grinding, coating hardness, determination of surface geometry, surface morphology, roughness, amount of work) were carried out after tests (estimation of wear marks) after tribological tests.

Tests were performed to determine the tribotechnical characteristics of samples without coatings with counterbodies (initial tests) and samples with the investigated Cu-Mo-N coatings with counterbodies. The results are given in table 2.

A study of friction pairs (surface morphology, roughness, amount of wear) was carried out after tribulation tests (geometric characteristics of wear marks (table 2).

Measured values of nanohardness and Young's modulus in Avinit-type coatings (based on Cu-Mo-N) using a device for measuring nanohardness of CSM (Switzerland) (load speed 20.00 mH/min, max depth 100.00 nm at load 0, 6 G) - K, = 800 - 900 Vickers, H = 900-1000 MPa, E = 190 -200 GPa, Poisson's ratio K = 0.30.

Tabele 2. Rating traces of wear on samples after tribological tests.

A pair of friction basic counter body	Absolutely load, kN	Coef. friction	No sample	Geometric characteristics traces of earnings			Notes
Samples without coating							
DIN 1.2379 Steel DIN 1.773, nitrided	0,2…1,4 at 0.8	0,02…0,067 0,11max	basic 023	Depth, μm	Width, mm	Roughness	production with rough relief and metal deposits
				180 max	5,85 max		
			counter body 6_{10}	Depth productions, mm			traces of adhesion with both deposits and metal tears
				There are no productions			
	0,2…0,8 at 0,4	0,04…0,115 0,03min	basic 023	Depth, μm	Width, mm	Roughness	production with rough relief and metal deposits
				150 max	5,15 max		
			counter body 6_{11}	Depth productions, mm			traces of adhesion with both deposits and metal tears
				There are no productions			
DIN 1.2379 Steel DIN 1.773, cemented	0,2…1,2	0,04…0,108	basic 1	Depth, μm	Width, mm	Roughness	production with rough relief and metal deposits
				80 max	4,51 max		
			counter body 7_4	Depth productions, mm			traces of adhesion with both deposits and metal tears
				There are no productions			
DIN 1.2379 Steel DIN 1.773, nitrided	0,2…1,4	0,07…0,086	basic 1	Depth, μm	Width, mm	Roughness	
				≈0,16	≈0,2	Ra0,044 (▼11в)	
			counter body 6_7	Depth productions, mm		Roughness	the friction surface has a dark coating
				There are no productions		Ra0,04 (▼11в)	
Coating of the main samples in the multilayer coating system based on Cu-(Mo-N)							
DIN 1.2379 Steel DIN 1.773, cemented	0,2…1,4 at 1.0	0,040…0,104 0,114max	Basic 031 coating: Single layer coating (Cu-Mo-N)	Depth, μm	Width, mm	Roughness	there is no coating inside the trace
				≈1,6	≈0,6	Ra0,04 (▼11в)	
			counter body 7_5	Depth productions, mm		Roughness	the friction surface has a dark coating
				There are no productions		Ra0,045 (▼11в)	
DIN 1.2379 Steel DIN 1.773, cemented	0,2…1,4 at 1.2	0,05…0,111 0,117max	basic 040 Nanolayered coating Cu-(Mo-N), ratio	Depth, μm	Width, mm	Roughness	
				≈1,6	≈0,6	Ra0,075 (▼11a)	

			of layer thicknesses -1:1; h≤1 μm				
			counter body 7_6	Depth productions, mm		Roughness	the friction surface has a dark coating
DIN 1.2379 Steel DIN 1.773, cemented	0,2…1,4	0,07…0,117	basic 040 Nanolayered coating Cu-(Mo-N), ratio of layer thicknesses -1:1; h≤1 μm	Depth, μm	Width, mm	Roughness	production with rough relief and metal deposits
				13max	1,4 max		
			counter body 7_2	Depth productions, mm			the friction surface has a dark coating
				There are no productions			
DIN 1.23791 Steel DIN 1.773, cemented	0,2…1,4	0,07…0,114	basic 042 Single layer coating (Cu-Mo), h≤1 μm	Depth, μm	Width, mm	Roughness	
				≈1,7	≈0,6	Ra0,03 (▼12б)	
			counter body $7_{б/н}$	Depth productions, mm		Roughness	the friction surface has a dark coating
				There are no productions		Ra0,04 (▼11в)	
DIN 1.2379 Steel DIN 1.773, nitrided	0,2…1,4	0,05…0,103	basic 042 Single layer coating (Cu-Mo), h≤1μm	Depth, μm	Width, mm	Roughness	
				≈1,8	≈0,6	Ra0,028 (▼12б)	
			counter body 6_8	Depth productions, mm		Roughness	the friction surface has a dark coating
				There are no productions		Ra0,026 (▼12б)	
DIN 1.2379 Steel DIN 1.773, cemented	0,2…1,4 at 1,0	0,034…0,114 0,147max	basic 048 Nanolayered coating Cu-Mo, ratio of layer thicknesses - 1:2, h≤1 μm	Depth, μm	Width, mm	Roughness	there are transverse lines, one of which can be seen in the traces of binding
				≈4	≈1,2	Ra0,04 (▼11в)	
			counter body 7_3	Depth productions, mm			the friction surface has a dark coating
				There are no productions			
DIN 1.2379 Steel DIN 1.773, nitrided	0,2…1,4 at 0,6	0,008…0,096 0,073min	basic 19 Microlayered coating Cu-Mo, ratio of layer thicknesses -2:1, h≤1 μm	Depth, μm	Width, mm	Roughness	production with rough relief and metal deposits
				180 max	5,9 max		
			counter body 6_9	Depth productions, mm			traces of adhesion with
				There are no productions			

							both deposits and metal tears
DIN 1.2379 Steel DIN 1.773, nitrided	0,2…1,6 at 0,6	0,1…0,054 0,113max	basic 23 Microlayered coating Cu-(Mo-N), ratio of layer thicknesses - 2:1, h≤1 μm	Depth, μm	Width, mm	Roughness	production with rough relief and metal deposits
				100 max	4,4 max		
			counter body 6_{12}	Depth productions, mm			traces of adhesion with both deposits and metal tears
				There are no productions			
DIN 1.2379 Steel DIN 1.773, cemented	0,2…1,2 at 0,4	0,09…0,06 0,12max	basic 23 Microlayered coating Cu-(Mo-N), ratio of layer thicknesses - 2:1, h≤1 μm	Depth, μm	Width, mm	Roughness	production with rough relief and metal deposits
				12	1,5		
			counter body 7_7	Depth productions, mm			slight traces of adhesion
				There are no productions			

3.2 Multi-layer coatings based on (Cu - C) (with different carbon content)

Tests were conducted to determine the tribotechnical characteristics of samples with counterbodies having the investigated coatings. The results are given in the table 3.

Fig. 1-6 show the time dependences of friction coefficients and temperature in the contact zone of tribosystems "Steel - Cu - C (I)", "Steel - Cu - C (II)", "Steel - Cu - C (III)", as well as the level wear of friction surfaces over time using the acoustic emission method [14].

Table 3. Tribotechnical characteristics of samples

№	Cube (coating)	Roller (coating)	Test results	
1	steel DIN 1.237, 56…61HRC	24CrMoV5-5 steel, nitriding, ≥700HV	50H (2min), Kfr =0,5; 100H (2 min), Kfr =0,213; 150H (2min), Kfr =0,18; 200H (2min), Kfr =0,168; 250H (2min), Kfr =0,167; 300H (2min), Kfr =0,149; 350H(2min),Kfr =0,14; 400H (2min), Kfr =0,133; 450H (2min), Kfr =0,128; 500H (2min), Kfr =0,127; 550H (2min), Kfr =0,126; 600H (2min), Kfr =0,12; 650H (2min), Kfr =0,116; 700H (2min), Kfr =0,11; 750H (2min), Kfr =0,109; 800H (2min), Kfr =0,104; 850H (2min), Kfr =0,102; 900H (2min), Kfr =0,099; 950H (2min), Kfr =0,096; 1000H (2min), Kfr =0,093; 1050H (2min), Kfr =0,092; 1100H (2min), Kfr =0,09;	The cube has a normal wear mark, minor metal deposits. Corresponding scratches on the roller

			1150H (2min), Kfr =0,093; 1200H – bully.	
2		Cu-C (I)	50H (2min), Kfr =0,4; 100H (2min), Kfr =0,28; 150H (2min), Kfr =0,22; 200H (2min), Kfr =0,19; 250H (2min), Kfr =0,15, the load was not increased further.	The coating on the roller is almost completely worn out. The cube has a normal wear mark, no wear.
3		Cu-C (II)	50H (2min), Kfr =0,49; 100H (2min), Kfr =0,28; 150H (2min), Kfr =0,2; 200H (1min), Kfr =0,2, the load was not increased further..	The coating on the roller is almost completely worn out. The cube has a normal wear mark, no wear
4		Cu-C (III)	50H (2min), Kfr =0,44; 100H (2min), Kfr =0,248; 150H (2min), Kfr =0,179; 200H (2min), Kfr =0,15; 250H (2min), Kfr =0,147; 300H (1,5min), Kfr =0,127, the load was not increased further	The coating on the roller is almost completely worn out. The cube has a normal wear mark, no wear

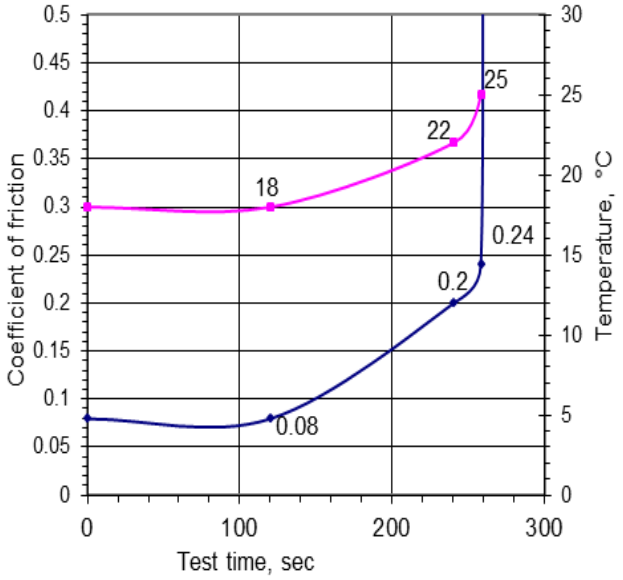


Fig. 1. Friction coefficient and temperature in the contact zone.

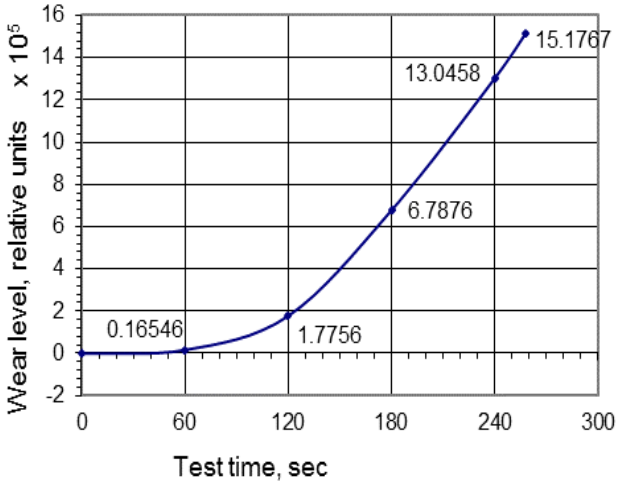


Fig. 2. The level of wear of friction surfaces Tribosystem steel – CuC (I).

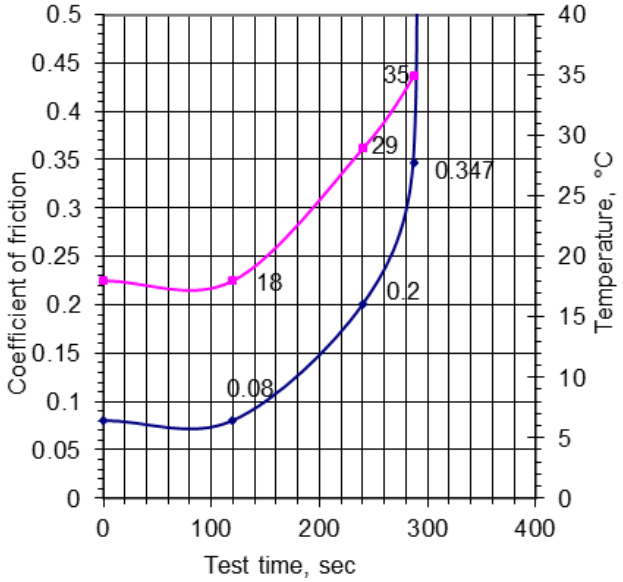


Fig. 3. Friction coefficient and temperature in the contact zone.

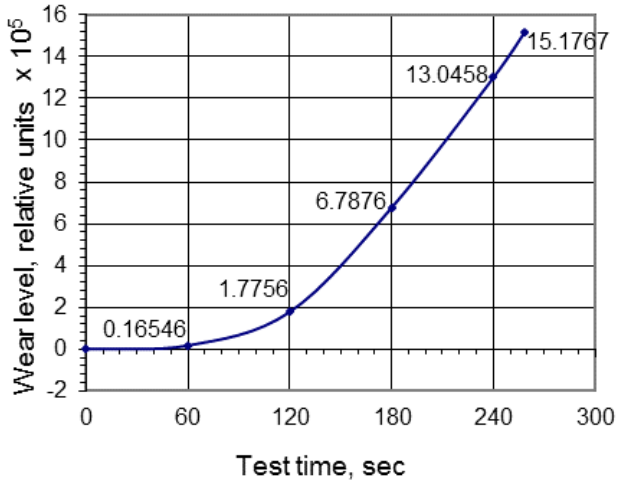


Fig. 4. The level of wear of friction surfaces Tribosystem steel – CuC (II).

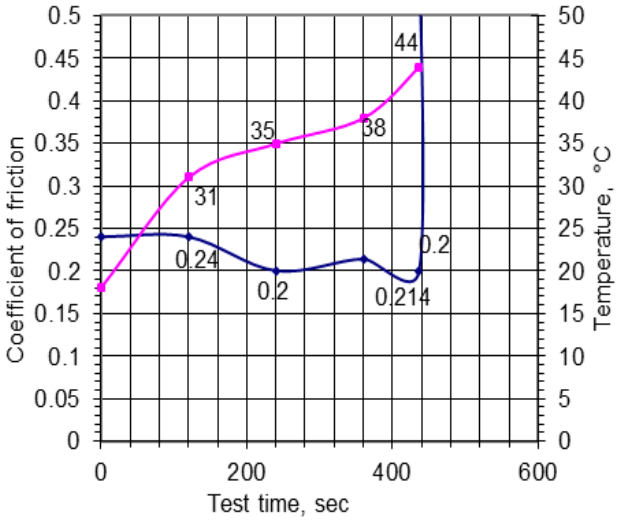


Fig. 5. Friction coefficient and temperature in the contact zone.

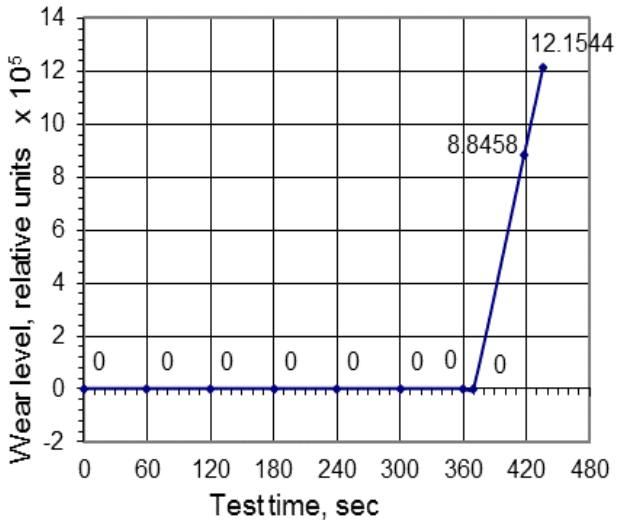


Fig. 6. The level of wear of friction surfaces Tribosystem steel – CuC (III).

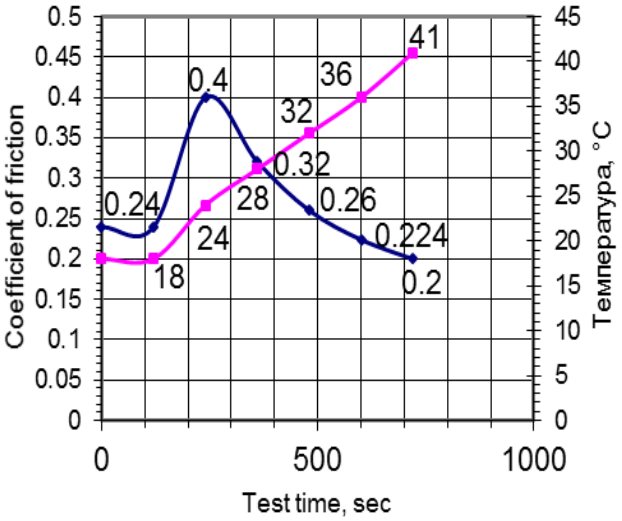


Figure 7. Friction coefficient and temperature in the contact zone tribosystem steel – steel.

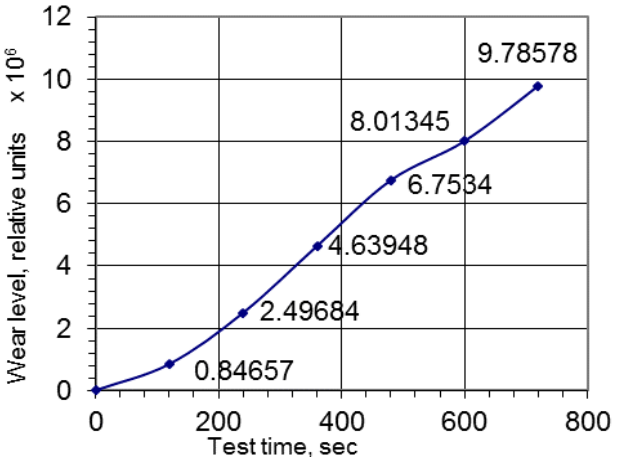


Fig. 8. The level of wear of friction surfaces.

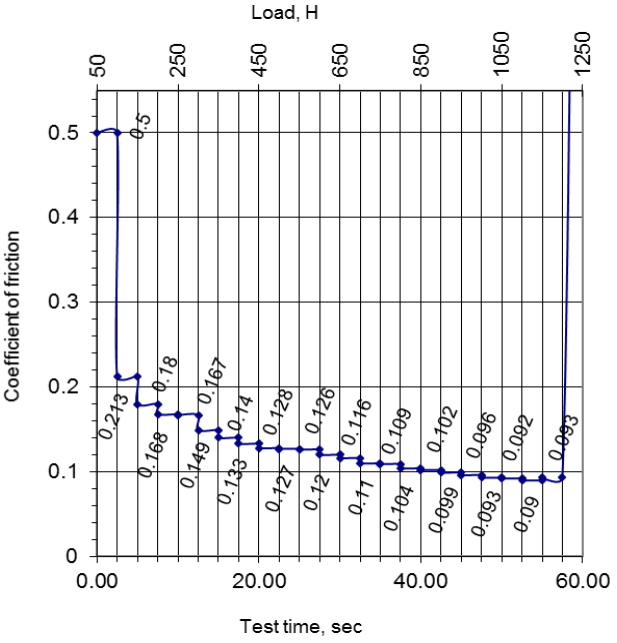


Fig. 9. Friction coefficient of the tribosystem (steel din 1.237) – (24crmov5-5). The maximum load is 1200 n.

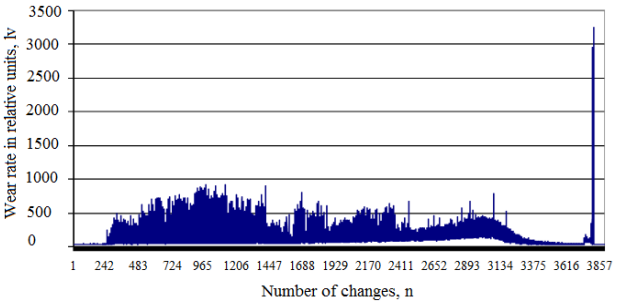
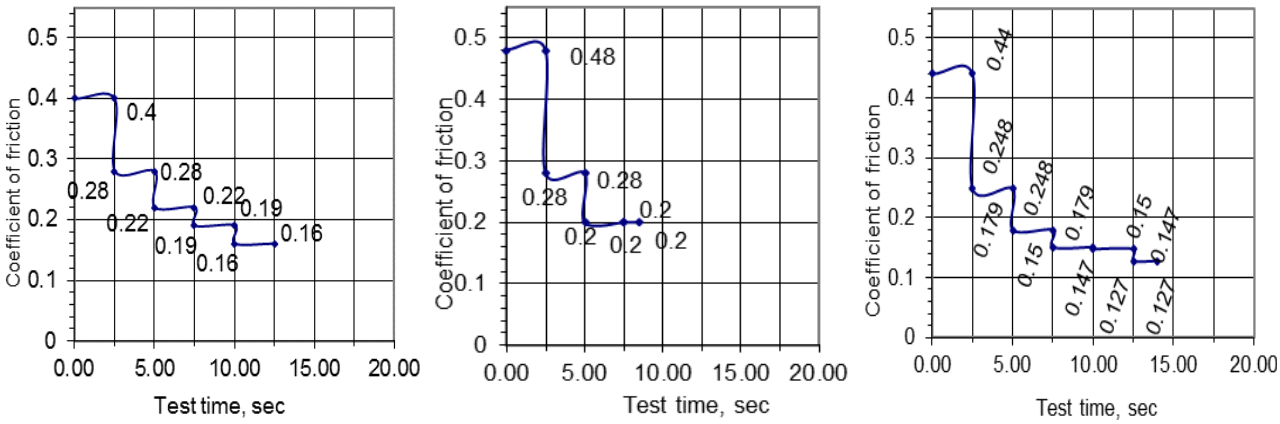


Fig. 10. The nature of the change in the rate of wear.

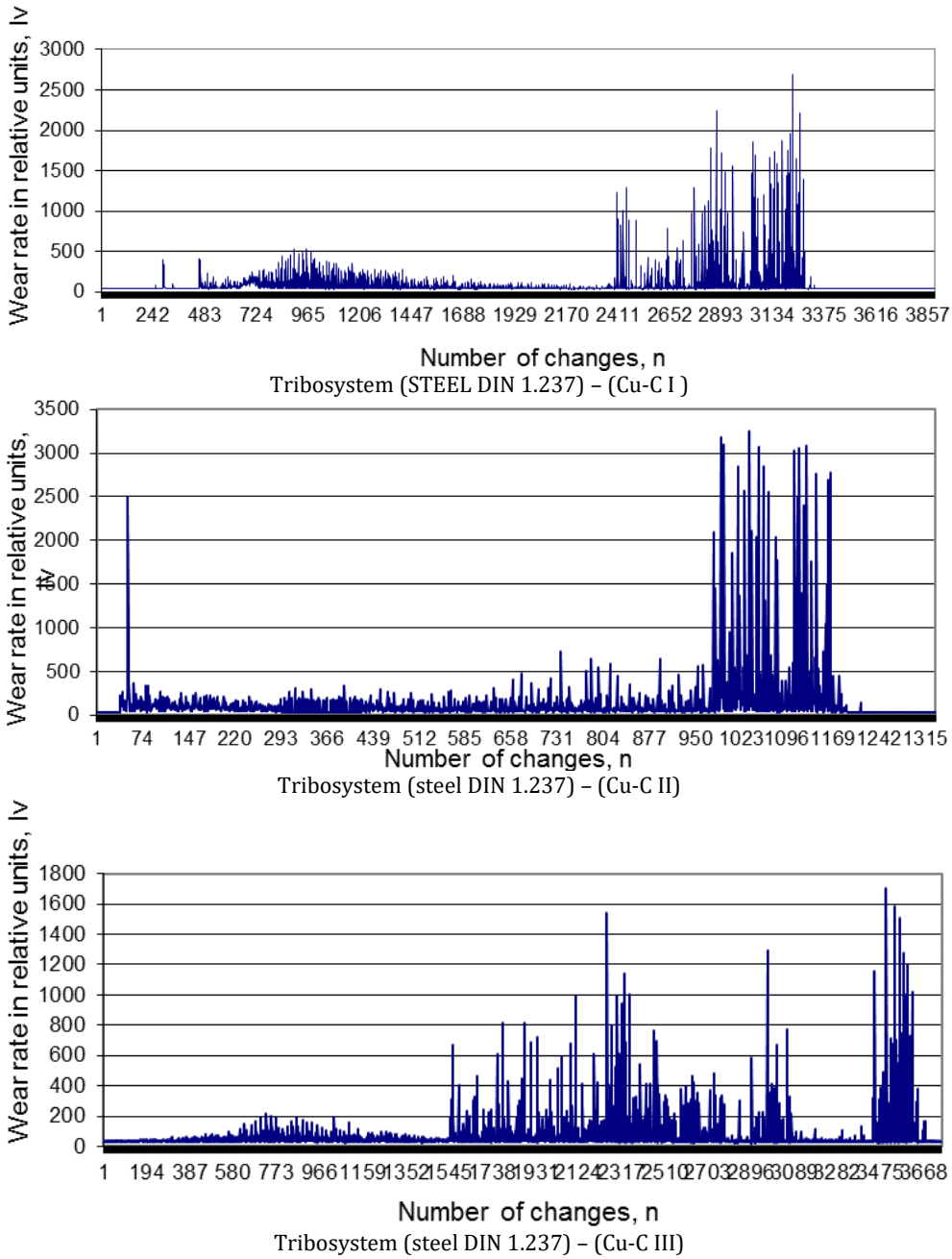
Step load of 50 N every 2 min. The maximum load is 250 N.

The test was stopped due to almost complete wear of the coating on the roller. The die has a mark of use without wear.



Tribosystem (steel din 1.237) – (Cu-C I). Tribosystem (steel din 1.237) – (Cu-C II) Tribosystem (steel din 1.237) – (Cu-C III)

Fig. 11. Friction coefficient of tribosystems (steel din 1.237) - (cu-c) over time.



Tribosystem (STEEL DIN 1.237) – (Cu-C I)

Tribosystem (steel DIN 1.237) – (Cu-C II)

Tribosystem (steel DIN 1.237) – (Cu-C III)

Fig. 12. The nature of the change in the rate of wear according to the time of the test.

4. CONCLUSION

Technological parameters of application of firmly bonded high-quality multi-layer and nano-layer coatings were worked out. At the same time, the following processes were worked out and "metal-metal nanocomposite coatings" were obtained:

- metal PVD coatings of Mo, Sg;
- multi-layer PVD coatings based on Cu-Mo-N;
- multi-layer PVD coated on a (Si-C) base (with different carbon content).

Samples with coatings were studied (micropolishing, coating hardness, determination of surface geometry after coating). Distortions of the geometry and roughness of the coated surfaces, compared to the condition before the coating, were not detected. The thickness of multilayer coatings is 1...2 microns. The measured values of the hardness of the coatings on the rollers were 250-400 kg/mm2 for the tested samples.

The developed regimes ensure high adhesion and preservation of the hardness of DIN 1.2379 steel within the specified limits.

Multi-layer and nano-layer coatings were obtained on the counterbodies (rollers) and on the main samples (cubes) for tribological tests.

Tribological tests of coated samples were carried out.

All tested coatings were completely worn out when rubbing steel DIN 1.2379 at very low loads (max up to 300N, then the load did not increase). All coatings had a high coefficient of friction Kfr ≥ 0.15.

The use of such coatings in tribocombinations with steel in the form of independent wear-resistant coatings is impractical. Obviously, they can be considered only as soft layers in the development of new designs of anti-friction wear-resistant coatings to increase the performance of friction pairs in the "coating - steel" and "coating - coating" systems.

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