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Influence of Magnetron Sputtering Parameters on Mechanical and Tribological Properties of Carbon Nitride Coatings

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This work is devoted to investigation of mechanical and tribological properties of carbon nitride (CN_x) coatings deposited on the titanium IM125 and steel AISI321 substrates by the method of reactive magnetron sputtering of a graphite target at direct current in a mixture of gases argon/nitrogen. As shown, at pressure $p = 0.35$ Pa, nitrogen concentration in a mixture of gases $C = 42$–58%, substrate temperature $T_{\text{substr.}} = 130$°C, the highest values of hardness $H = 15$–20 GPa, modulus of elasticity $E^* = 120$–132 GPa, normalized hardness $H/E^* = 0.130$–0.152, elastic deformation $\varepsilon_{es} = 4.03$–4.6% are obtained, indicating increased ductility and wear resistance of the coating under friction. Wear-resistance testing conducted under dry friction by means of the ball–on–disc and ball–section methods show that high wear resistance is found in CN_x coatings with the highest carbon ordering, and it is confirmed by high values of normalized hardness H/E^* and ε_{es}. Testing in blood plasma of titanium IM125 samples with CN_x coating in friction pair with chirulen yields low values of friction coefficient and wear intensity of chirulen equal to 0.079 and 0.65 mm/km, respectively.

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Key words: reactive magnetron sputtering, carbon nitride coatings, microindenting, tribological properties, graphite target, biomedical compatibility.

Дану роботу присвячено дослідженню механічних і трибологічних властивостей покриттів нітриду Карбону CN_x, нанесених на основи з титану ІМ125 і криці AISI321 методом реактивного магнетронного розпорошення графітової мішені на постійному струмі в суміші газів арґон/азот. Показано, що за тиску $p = 0,35$ Па, концентрації азоту в суміші газів $C = 42–58\%$, температурі основи $T_{основи} = 130°C$ одержано найбільші значення твердости $H = 15–20$ ГПа, модуля пружности $E^* = 120–132$ ГПа, нормованої твердости $H/E^* = 0,130–0,152$, пружньої деформації $\varepsilon_{es} = 4,03–4,6\%$, що свідчить про підвищену пластичність і зносостійкість покриття за тертя. Випробування зносостійкости, проведені в умовах сухого тертя методами куля–диск і куля–шліф, показали, що підвищену зносостійкість виявлено у покриття CN_x з найвищим карбоновим упорядкуванням, що підтверджується високими значеннями нормалізованої твердости H/E^* і показником пружньої деформації ε_{es}. У дослідженні в плазмі крові зразків титану ІМ125 з покриттям CN_x у парі тертя з хіруленом встановлено низькі значення коефіцієнта тертя й інтенсивности зношування хірулена, які дорівнювали 0,079 і 0,65 мм/км відповідно.

Ключові слова: реактивне магнетронне розпорошення, покриття нітридом Карбону, мікроіндентування, трибологічні властивості, графітова мішень, біомедична сумісність.

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1. INTRODUCTION

As shown theoretically, carbon nitride (CN_x) should be harder than diamond, if it has β-C_3N_4 structure [1]. Even though CN_x coatings still do not have such an ideal theoretical structure, its high hardness in the range of 15–50 GPa was obtained by several research groups [2–4]. Compared with hydrogenated diamond like carbon coatings, CN_x has a higher wear resistance at a low friction coefficient [5]. Tribological studies of CN_x coatings of 100 nm thick, deposited by ion-beam method on silicon plates, revealed that the lowest friction coefficient is found in nitrogen, CO_2, and vacuum ($\mu = 0.01–0.1$), and the highest—in air and oxygen ($\mu = 0.2–0.4$). Friction coefficient is decreased after a certain number of friction cycles. Particles of CN_x wear appear on the interphase of two sliding surfaces, from which agglomerates, moving layers and layer mixtures form. Some of them promote matching of the two surfaces, other components on the contact interphase act as a good lubricant [6]. It is found that both mechanical and physic-chemical properties of CN_x layers depend on their nitrogen content: biocompatibility, wetting with synovial fluid in human plasma. Owing to these properties, CN_x coating can be used in end prostheses and orthodontic

[7–12].

CN_x coatings are mainly produced by the method of reactive magnetron sputtering of a graphite target in a mixture of gases Ar/N_2. For instance, [13] gives the results of studying mechanical and tribotechnical properties of CN_x magnetron coatings 0.5 μm thick, which are deposited on silicon, nickel, and tool steel substrates at changing content of N_2 and substrate temperature of 100°C and 350°C. As shown, the maximum value of elastic restoration of the coating surface, characterizing its increased wear resistance, reaches $W_e = 78–90\%$ in $CN_{0.19}$ and $CN_{0.25}$ coatings; wear rate of coatings deposited at $T = 350°C$ is two times higher.

The paper presents the results of investigation of mechanical and tribological properties of CN_x magnetron coatings ($\delta = 2–4$ μm).

2. EXPERIMENTAL/THEORETICAL DETAILS

Coatings were deposited using upgraded vacuum unit VU-1BS, which was fitted with direct current magnetron sputtering module, consisting of two magnetrons: Magnetron 1 with disc MPG-7 graphite (99.98% purity) target (∅ = 88 mm, 4 mm thickness) and Magnetron 2 with rectangular IM125 titanium alloy target (90×58×4 mm). Magnetrons were mounted so that the angle between target surfaces was equal to 150°. This enabled simultaneous or alternative deposition of coatings on a stationary substrate from two magnetrons at the same distance between the substrate and targets equal to 110 mm. Magnetron 2 was designed for deposition of adhesion bond coat of titanium and TiCN interlayer.

The following were used as substrates: a) AISI321 steel and IM125 titanium samples of 65×30×0.5 mm size, b) IM125 titanium sample of ∅ = 25 mm and $\delta = 5$ mm.

Before placing into the vacuum chamber, the samples were cleaned in an ultrasonic bath, successively filled with acetone and ethyl alcohol. At $p = 5.0 \cdot 10^{-4}$ Pa, the sample was preheated at $T_{substr.} = 150°C$ for 20 min, then without switching the heater off the sample surface was cleaned by bombardment with argon (high purity) ions in a direct current glowing discharge at $p = 1.3$ Pa, $U = 1100$ V for 20 min.

CN_x coatings formed on the sample surface through a titanium bond coat and TiCN interlayer at the following parameters:

a) deposition of titanium bond coat ($\delta = 0.3$ μm) in Ar at $p = 0.35$ Pa, specific power of magnetron discharge with titanium target $\Delta = 3.5$ W/cm, deposition rate $V_{Ti} = 25$ nm/min, substrate bias voltage U varying from −300 V up to −1400 V;

b) deposition of intermediate layer of TiCN ($\delta = 0.13–0.25$ μm) using joint reactive magnetron sputtering of the graphite and titanium targets at direct current in a mixture of gases Ar/N_2 at $p = 0.35$ Pa, 1 Pa and 2 Pa, volume concentration of nitrogen in Ar/N_2 mixture

$C = 25.6\%$, ratio of powers of magnetron discharge at targets $P_C/P_{Ti} = 2.7–3.0$, $U = 0$–-40 V;

c) deposition of CN_x main layer ($\delta = 2–3.9$ μm) in the mixture of gases Ar/N_2 at $p = 0.35$ Pa, 1 Pa and 2 Pa, $C = 42–58\%$, $\Delta = 10$ W/cm, $U = 0$–-40 V, $T_{substr.} = 130, 200, 350$°C.

The investigation of influence of magnetron sputtering process parameters on phase composition and structure of carbon nitride coatings is presented in [14].

Mechanical testing of three-layer CN_x coatings was performed by microindentation method with Micron-Gamma instrument. Parameter values were calculated automatically by ISO 14577-1:2002 standard.

Evaluation of abrasion wear resistance under dry friction of CN_x coatings, deposited on IM125 titanium samples ($\delta = 6$ mm, $\varnothing = 25$ mm) was performed by the methods of ball–on–disc and ball–section. Tribological testing of IM125 titanium samples with CN_x coating in friction pair with chirulen (ultra-high molecular weight polyethylene—UHMW PE) in blood plasma was also performed.

Determination of coating wear resistance by the method of ball–on–disc was conducted using an instrument consisting of an electromechanical drive, stage for sample fastening, rotating in the horizontal plane, indenter with replaceable steel ball with $\varnothing = 5.5$ mm. Diameter of the track of coating abrasion varied in the range of 10 to 18 mm by shifting the indenter axis relative to that of stage rotation. Coatings were tested for 60 minutes at the 2 N and 3.5 N loading on the indenter. Coating wear resistance was assessed using MBS-8 microscope by the appearance of the annular track crater, formed on the length of the covered path L_p at the number of rotations $n = 2000$, 2500, 3000. Coating was considered more wear-resistant at smaller width and depth of the track crater, at smaller number of delaminations on the track. The width and depth of the track crater and dimensions of coating delamination were measured by differentiated profilometer Micron-alpha.

Determination of wear resistance by ball–section method was conducted using an instrument ensuring contact impact of the rotating ball of $\varnothing = 23.8$ mm on a flat sample; a crater of semi-spherical shape formed on the coating. Crater diameter $\varnothing_{crater.}$ and depth $h_{crater.}$ were measured with an interference profilometer Micron-alpha. Coating was considered more wear-resistant at smaller crater diameter.

Testing of CN_x coatings in blood plasma was performed in a faceplate friction machine (Fig. 1) (according to ASTM F732-82) at the V. Bakul Institute for Superhard Materials of the National Academy of Sciences of Ukraine. Sample ($\varnothing = 21.5$ mm, $R_a = 54–69$ nm) with CN_x coating (Fig. 2) was pressed by force P to fixed chirulen counterbody, that resulted in formation of a traction pair of IM125 titanium—CN_x + chirulen. The counterbody was a cylinder of $\varnothing = 52$ mm and

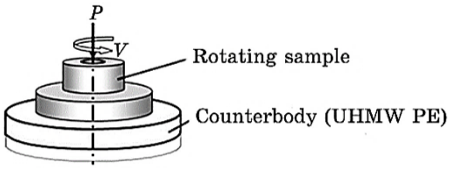

Fig. 1. Scheme of testing in faceplate friction machine.

Fig. 2. IM125 titanium samples with CN_x coating for tribological testing.

15 mm height, it is working surface roughness being $R_a = 3$ μm. Speed of sample sliding along the counterbody was equal to $V = 0.057$ m/s, contact pressure was 3.5 MPa. Form and depth of wear crater on the counterbody surface and chirulen wear rate was determined by the profilogram, recorded in VEI Kalibr instrument. During testing, the oscillogram of the torque change was also recorded, which was the base for calculation of the change of friction coefficient f in time.

3. RESULTS AND DISCUSSION

Results of mechanical testing of samples with CN_x coatings are shown in Table 1 and Table 2; there are depending of coatings mechanical properties (hardness H, modulus of elasticity E^*, normalized hardness H/E^*, elastic deformation ε_{es}) on sputtering parameters, substrate materials and conditions of ion bombardment. Analysis of the results shows that:

a) the highest values of hardness $H = 15$–20 GPa, modulus of elasticity $E^* = 120$–132 GPa, normalized hardness $H/E^* = 0.130$–$0.152 > 0.12$ were obtained on titanium substrates at $p = 0.35$ Pa, $T_{substr.} = 130$°C and $C = 42$–58% that is indicative of increased ductility and wear resistance of the coating under friction ($6CN_x$, $8CN_x$ samples);

b) at increase of substrate temperature up to 350°C, coating hardness is reduced two times;

c) the highest values of hardness $H = 15$ GPa, normalized hardness $H/E^* = 0.124$, elastic deformation $\varepsilon = 3.28$% were obtained at ion

TABLE 1. Mechanical properties of CN_x coatings, deposited on AISI321 steel and IM125 titanium substrates.

Sample No.	Substrate material	Sputtering parameters			Mechanical properties			
		Pressure p, Pa	Nitrogen content CN_2, %	$T_{substr.}$, °C	H, GPa	E^*, GPa	H/E^*	ε_{es}, %
$1CN_x$	AISI321	0.35	58	130	10.1	96	0.105	3.21
$2CN_x$	AISI321	0.35	100	130	6.0	95	0.063	1.93
$3CN_x$	AISI321	0.35	24	130	4.8	95	0.051	1.6
$4CN_x$	AISI321	1.0	23	130	3.8	80	0.048	1.45
$5CN_x$	AISI321	1.0	61	130	1.5	74	0.02	0.64
$6CN_x$	IM125	0.35	58	130	15.6	120	0.130	4.027
$7CN_x$	IM125	0.35	58	350	7.0	110	0.064	1.95
$8CN_x$	IM125	0.35	42	130	20.0	132	0.152	4.634

TABLE 2. Mechanical properties of CN_x coatings, depending on the conditions of ion bombardment (IM125 titanium substrates).

Sample No.	Sample R_a, nm	Conditions of ion bombardment		Mechanical properties			
		Bias voltage, $U_{b.Ti}$, V	Bias voltage, $U_{b.CN_x}$, V	H, GPa	E^*, GPa	H/E^*	ε_{es}, %
$9CN_x$	65	−1000	0	15	121	0.124	3.28
$10CN_x$	160	−1400	−30	8.2	108	0.076	2.30
$11CN_x$	120	−1400	−10	10	108	0.093	2.81
$12CN_x$	145	−1400	−40	8.7	112	0.078	2.36
$13CN_x$	50	−1000	−10	10.8	100	0.108	3.28
$14CN_x$	50	−1000	−10	5.6	79	0.071	2.15

treatment of the coating ($U_{b.Ti} = -1000$ V and $U_{b.CN_x} = 0$), indicating a greater proneness of CN_x coating to stress relaxation ($9CN_x$ sample);
d) increasing of bias voltage at deposition of a titanium bond coat, intermediate TiCN layer and CN_x layer ($U_{b.Ti} = -1400$ V, $U_{b.CN_x} = -30$–-40 V) leads to lowering of all the parameters H, E^*, H/E^*, ε_{es}.

The main parameter determining the quality of produced CN_x coatings is their wear resistance under dry friction. Therefore, dependences of coating wear resistance on i) thickness of intermediate interlayer and main layer and ii) its deposition conditions were assessed (Tables 3, 4).

TABLE 3. Dependence of wear resistance of CN_x coating on the thickness of intermediate and main layers (titanium IM125 substrate).

Sample No.	Sample R_a, nm	Layer thickness			Wear resistance (Testing method 'ball–on–disc')
		δ_{Ti}, μm	$\delta_{Ti–C–N}$, μm	δ_{CN_x}, μm	
18CN_x	49 ± 7	0.3	0.25	1.8	$P_{ind.}$ = 2 N. No CN_x delaminations on the track. $P_{ind.}$ = 3.5 N. Spot CN_x delaminations on 80% of the track.
19CN_x	87.5 ± 22	0.3	0.25	3.6	$P_{ind.}$ = 2 N. No CN_x delaminations on the track. $P_{ind.}$ = 3.5 N. Spot CN_x delaminations on 20% of the track.
20CN_x	57.5 ± 17.5	0.3	0.13	1.8	$P_{ind.}$ = 2 N. Spot and dotted delaminations on 70% of the track.

TABLE 4. Dependence of wear resistance of CN_x coating on the thickness of intermediate and main layers (titanium IM125 substrate).

Sample No.	p, Pa	$T_{subtr.}$, °C	Sputtering parameters						Wear resistance (Testing method 'ball–on–disc', $P_{ind.}$ = 3.5 N)
			Ti		Ti–C–N		CN_x		
			P_{Ti}, W	$U_{b.}$, V	CN_2, %	P_C/P_{Ti} rel. units	CN_2, %	P_C, V	
15CN_x	0.35	200	184	–150	25.6	2.7	58	570	n = 2000; L_p = 94 m Spot delaminations on 60% of the track
6CN_x	0.35	130	184	–150	25.6	2.8	58	560	n = 3000; L_p = 174 m No crater, coating in the track is rolled.
16CN_x	0.35	130	184	–300	–	–	58	580	n = 2500; L_p = 99 m Spot delaminations on 20% of the track
8CN_x	0.35	130	184	–300	25.6	2.8	42	540	n = 3000; L_p = 174 m No crater, coating on the track is rolled
17CN_x	0.35	350	180	–300	25.6	2.8	42	540	n = 2000; L_p = 95 m Spot delaminations on 50% of the track

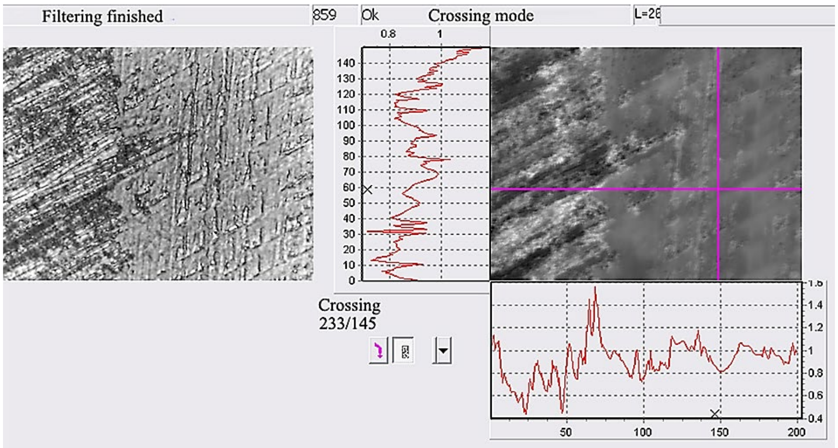

Fig. 3. Profilogram of CN_x coating on $6CN_x$ sample and half-width of the track obtained at testing by ball–on–disc method.

Analysis of the results given in Tables 3 and 4 shows the following.

a) At increase of the thickness of main CN_x coating, its wear resistance is increased (see comparison of tracks on samples $19CN_x$ and $18CN_x$ at $P_{ind} = 3.5$ N).

b) At decrease of the thickness of TiCN intermediate layer from 0.25 to 0.13 µm, coating wear resistance markedly decreases (see samples $18CN_x$ and $20CN_x$). Therefore, the thickness of TiCN layer should be equal to 0.25 µm.

c) Highest wear resistance of CN_x coating was obtained at $p = 0.35$ Pa, $T_{substr.} = 130°C$, $C = 58\%$ and 42% (on $6CN_x$ and $8CN_x$ samples no crater formed on the track at friction path length $L_p = 145$ m).

Figure 3 gives the profilogram of half of the track width on the coating of $6CN_x$ sample, recorded using an interference profilometer Micron-alpha. Coating on the track is rolled; surface roughness on it is four times lower, compared to the total coating roughness.

Dependence of CN_x coating wear resistance on ion treatment conditions was also studied (Table 5). Before producing coatings on samples specified in Table 5, roughness of substrate surface was measured. Coatings were formed at the following parameter values: $p = 0.35$ Pa, $T_{substr.} = 130°C$, $C = 42\%$.

Analysis of the data given in Table 5 leads to the following conclusions.

1. Wear resistance of CN_x coating depends on substrate surface roughness and ion bombardment conditions: wear resistance decreases with roughness reduction.

2. The most wear-resistant CN_x coating was produced on the sample with higher roughness. So, at coating testing on $11CN_x$ sample

TABLE 5. Dependence of wear resistance of a three-layer coating on the conditions of ion treatment and roughness of substrate surface (titanium IM125 substrates).

Sample No.	Sample R_a, nm	δ_{CN_x}, μm	Ion treatment conditions		Methods of wear resistance determination		
			$U_{b.Ti}$, V	$U_{b.CN_x}$, V	'Ball–on–disc', $P_{ind.} = 3.5$ N	'Ball–section'	
						$T_{crater.}$ μm	$h_{crater.}$ μm
18CN$_x$	49	1.8	–300	0	Spot delaminations on 80% of the track	414	2
9CN$_x$	65	1.8	–1000	0	No delaminations on the track	510	2.1
10CN$_x$	160	2.0	–1400	–30	No delaminations on the track	400	1.9
11CN$_x$	120	2.1	–1400	–10	No delaminations on the track	310	1.7
12CN$_x$	145	2.1	–1400	–40	Spot and dotted delaminations on 50% of the track	414	2
13CN$_x$	50	2.2	–1000	–10	Dotted delaminations and rubbing off the coating on 60% of the track	560	2.7

($R_a = 120$ nm), produced at $U_{b.Ti} = -1400$ V and $U_{b.CN_x} = -10$ V there are no coating delamination's on the track, and crater diameter is minimum and equal to 310 μm.

3. At optimum roughness ($R_a = 65$ nm), the highest wear resistance of the coating is achieved at the following values of bias voltage: $U_{b.Ti} = -1000$ V and $U_{b.CN_x} = 0$ V (9CN$_x$ sample, Fig. 4).

4. With increasing of bias voltage applied to the substrate at deposition of intermediate and main layers, internal stresses in the coating become higher that leads to adhesion lowering. This is confirmed by testing the coating, which was formed on 12CN$_x$ sample ($R_a = 140$ nm) at $U_{b.CN_x} = -40$ V.

Conducted tribological testing showed that higher wear resistance of CN$_x$ coating is confirmed by high values of normalized hardness $H/E^* = 0.130 > 0.12$, elastic deformation percent $\varepsilon_{es} = 4.027$ and orderliness of carbon in the amorphous structure (small ratio of intensities of the Raman scattering bands $I_D/I_G = 1.20$ relative units [14]).

Therefore, wear resistant CN$_x$ magnetron coating can be obtained at the speed of 2.2 μm/hour on titanium IM125 and steel AISI321 substrates, when fulfilling the following technological requirements:
a) maintaining sample heating temperature $T_{substr.} = 130$–150°C during the entire process of coating formation at $p = 0.35$ Pa;

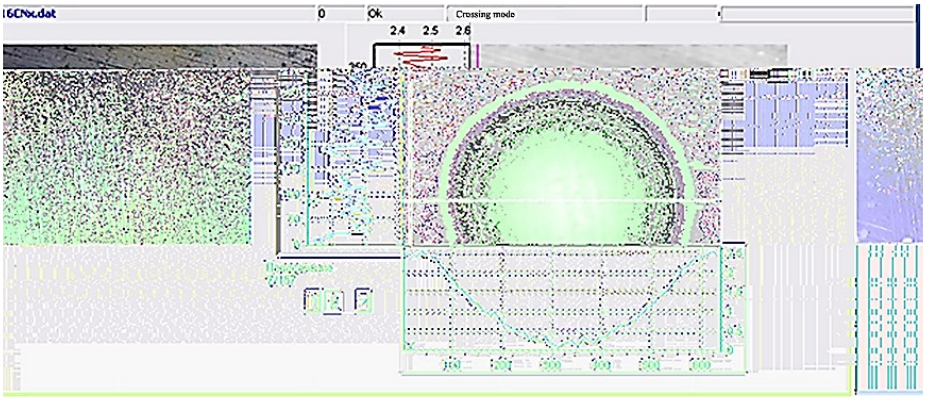

Fig. 4. Profilogram of a crater obtained on CN_x coating of $9CN_x$ sample at testing by ball–section method.

b) adhesion bond coat deposition Ti ($\delta = 0.3$ μm): $\Delta = 3.5$ W/cm, $C = 0\%$, $U_{b.Ti} = -1000$ V at $t_1 = 4$ min., $U_{b.Ti} = 0$ V at $t_2 = 10$ min;
c) deposition of intermediate TiCN layer ($\delta = 0.25$ μm): $\Delta = 10$ W/cm, $\Delta = 3.5$ W/cm^2, $U_{b.CN_x} = 0$ V, $C = 25.6\%$, $t = 10$ min;
d) deposition of the main CN_x layer ($\delta = 3$ μm): $\Delta = 10$ W/cm, $C = 58\%$, $U_{b.CN_x} = 0$ V.

Tribological testing of samples of IM125 alloy with CN_x coating (in friction pair with chirulen) in blood plasma showed that at the beginning of friction path value of friction coefficient was equal to $f = 0.17$–0.29. On the friction path of 0.472–0.627 km, fiction coefficient decreased 2–3 times, reaching the value of $f = 0.079$–0.09 and further on it did not change as a result of formation of a stable tribolayer on the coating, consisting of a large number of smooth pockets on the coating surface, accommodating the blood plasma. Results of tribological testing in blood plasma of CN_x coating are shown in Table 6.

According to the functional characteristics of the friction pair, the CN_x-coating/chirulen significantly exceeds the Co–Cr–Mo/chirulen pair traditionally used in the practice of endoprosthesis. The coeffi-

TABLE 6. Results of tribological testing in blood plasma of IM125 titanium samples with CN_x coating in friction pair with chirulen.

Sample No.	δ_{CN_x}, μm	Friction coefficient, f	Intensity of chirulen wear, mm^3/km
$6CN_x$	3.7	0.09	0.7
$7CN_x$	2.6	0.079	0.65
$8CN_x$	3.7	0.08	1.1

cient of friction in it is lower in 1.3 times and the wear of chirulen is 3.3 times lower [15].

4. CONCLUSIONS

1. Mechanical properties of CN_x coating ($\delta = 2$–4 μm) were studied. It is found that the highest values of hardness $H = 15$–20 GPa, modulus of elasticity $E^* = 120$–132 GPa, elastic deformation $\varepsilon_{es} = 4.03$–4.6% and normalized hardness $H/E^* = 0.130$–$0.152 > 0.12$ were obtained on titanium substrates at $p = 0.35$ Pa, $T_{substr.} = 130$°C and nitrogen concentration in the mixture of gases $C = 42$–58% that is indicative of increased ductility and wear resistance of the coating under friction.
2. Tribological testing by ball–on–disc and ball–section methods under dry friction showed that wear resistance of CN_x coating depends on: substrate surface roughness; values of parameters $T_{substr.}$, p, C, providing $H/E^* \geq 0.12$; thickness of intermediate TiCN layer; values of bias voltages $U_{b.Ti}$ and $U_{b.CN_x}$, applied at deposition of Ti bond coat, TiCN and CN_x layers.
3. Tribological testing of IM125 titanium samples with CN_x coating in a friction pair with ultra-high molecular weight polyethylene (chirulen) was conducted in blood plasma. It was determined that the friction coefficient and wear intensity of chirulen are equal to 0.079 and 0.65 mm³/km. Obtained intensity of chirulen wear is the lowest, compared to friction pairs, where titanium strengthened by other methods were used, and is 3.3 times lower than in the widely applied Co–Cr–Mo/chirulen pair.
4. Owing to high wear resistance and elasticity, CN_x coating can be recommended for CN_x deposition on interacting surfaces of items in the medical and instrument-making industry.

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