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NANOSTRUCTURAL SEPARATED TiN-BASED PROTECTIVE COATINGS

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Abstract: The results of the investigations of the physical mechanical and operational characteristics of the multicomponent (Ti,Zr)N coatings, generated by physical vapor deposition from the separated plasma flows are presented.

Keywords: separation, multicomponent coatings, microhardness, friction factor, adhesion strength, wear-resistance.

1. INTRODUCTION

Nowadays the development of new ways of different materials surface properties improving is the main direction in progress of the modern mechanical engineering, motor-car construction and woodworking industry. The surface of the cutting tools, machines and mechanisms is known to be exposed to different intensive destruction impacts [1]. Therefore, one of the promising ways of the products long-life increasing is their wear-resistance improvement, and it can be achieved by plasma technologies of the surface modification, allowing to control the composition, structure and physical mechanical properties of the materials surface layers.

2. MATERIALS AND METHODS

The modernized vacuum-arc plant of the wear-resistance multicomponent coatings deposition, equipped by Y-shaped plasma filter and magnetic solenoid system was applied in this work [2]. The zirconium and titanium vacuum-arc evaporators were used for the multicomponent coatings deposition. The element composition was controlled by the zirconium arc current changing (40–80 A), while the other technological parameters (titanium arc current, substrate bias, partial nitrogen pressure, deposition time) were constant.

The morphology and structure of the deposited coatings were studied by means of transparent electron microscope S–4800 Hitachi. The element concentration in the coatings composition was determined by the microanalyzer JXA–8500F. The phase composition of the multicomponent coatings were studied by X-ray diffractometer ДРОН–3М in the Cu–Kα radiation (λ=0,15418 nm).

Microhardness was measured by microindenter Duramin under 0.25N load. The friction factor of the deposited coatings was measured by means of the tribo-test device in the open air without any lubricant (counter body HB = 200 MPa). Adhesion strength was determined by scratch-tester JLST022 (J&L Tech). The specific volume wear was chosen to be as a parameter, characterizing the coatings wear and it was calculated according to [3].

3. RESULTS AND DISCUSSIONS

The transmission efficiency of the separated system was determined by the relation of the separator exit material mass to the separator entrance material mass (table 1).

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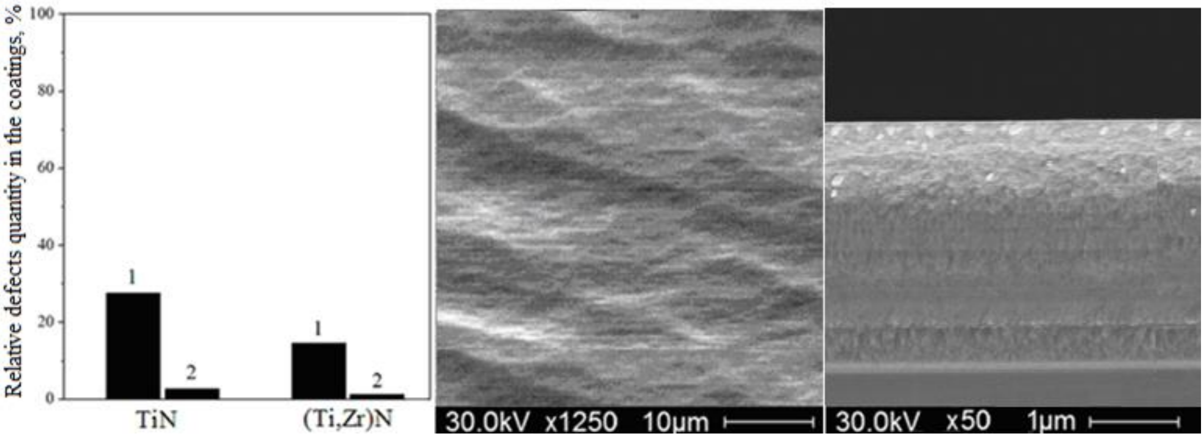
Table 1. The transmission efficiency of the Y–shaped plasma filter.

Coating	I_{Ti}, A	$I_{Zr.}$, A	Coating mass, mg		Transmission efficiency, %
			Separator entrance	Separator exit	
(Ti,Zr)N	60	40	17,7	2,1	11,9
		60	20,8	2,5	12,0
		80	23,1	2,8	12,1

The transmission efficiency for the multicomponent coatings (Ti,Zr)N has little influence on the arc current discharge due to practically erosion cathodes rate equality of the titanium and zirconium and is approximately 12%.

The separation system application allowed to decrease 1–5 µm sized defects of the deposited coatings up to 5,6 times for TiN and up to 4,3 times for (Ti,Zr)N (figure 1 a). The morphology investigation of the deposited coatings showed, that the coatings surface is characterized by the fine-grained structure (figure 1 b, c).



a b c

Figure 1. Diagram of the defects distribution in the coatings (*a*), surface morphology (*b*) and structure (*c*) of the deposited coatings (1 – deposition without plasma filter; 2 – deposition with plasma filter).

As a result of carried out experiments is found, that solid solution $Ti_{1-x}Zr_xN$ on the base of cubic lattice of structural type NaCl is the main crystal component for the (Ti,Zr)N coatings in no dependence on the zirconium concentration. The reflection intensity from the crystal plane (111) has a clearly-expressed peak for the multicomponent coatings (Ti,Zr)N at all zirconium concentrations. Lattice parameter of (Ti,Zr)N coatings rises from 0,4288 to 0,4456 nm with zirconium concentration increase, since zirconium atomic radius (0,160 nm) is higher than titanium atomic radius and the $Ti_{1-x}Zr_xN$ solid solution formation on the base of TiN takes place. It's found, that the addition of the alloying element (zirconium) in the TiN coating leads to the crystallite size decrease to 6–9 nm for the investigated zirconium concentration range (4–21 at. %) of the multicomponent coatings (table 2).

Table 2. Structural characteristics of the multicomponent coatings in the dependence of the technologic parameters.

Coating	$P \cdot 10^{-2}$, Pa	I_{Zr}, A	I_{Ti}, A	Zr, at. %	Ti, at. %	N, at. %	*a*, nm	*D*, nm
(Ti,Zr)N	3,0	40	60	4,34	46,91	48,75	0,4288	8,8
		50		7,82	42,95	49,23	0,4326	7,6
		60		11,71	38,15	50,14	0,4348	6,9
		70		17,16	32,92	49,92	0,4432	6,5
		80		21,71	28,22	50,07	0,4456	6,4
TiN		–	60	–	48,23	51,77	0,4245	37,8

The microhardness of the multicomponent (Ti,Zr)N coatings heightened up to 37 GPa with zirconium concentration increasing (figure 2), and it’s attributed to the grain structure fragmentation of the coating material.

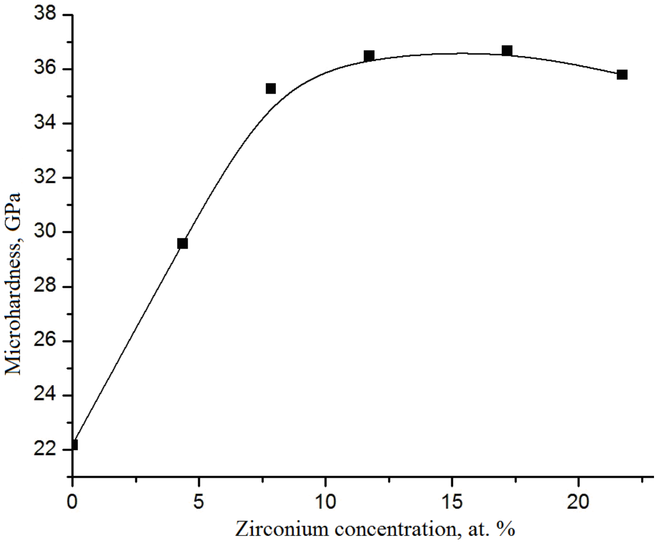
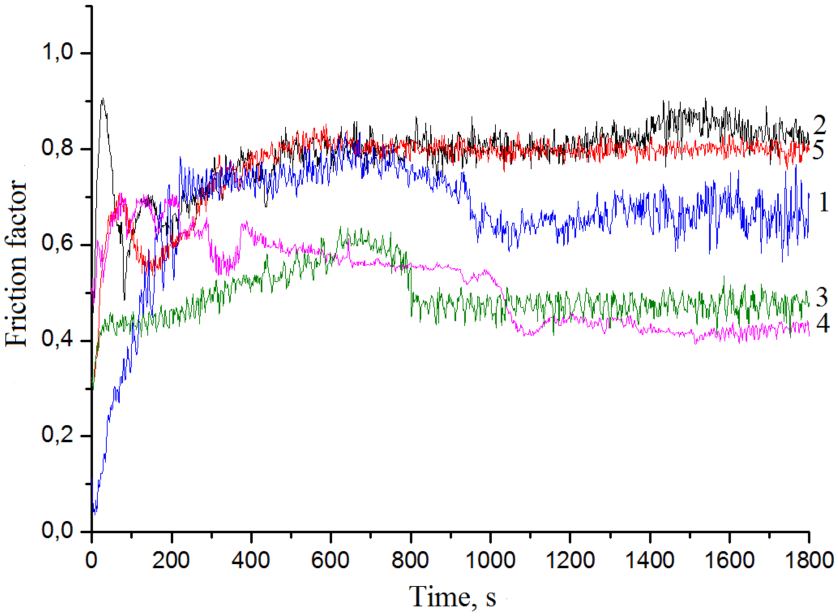


Figure 2. Dependence of the microhardness of the (Ti,Zr)N coatings on the zirconium concentration.

The friction factor investigations of the (Ti,Zr)N coatings under different zirconium concentration allowed to find out its optimal concentration range in the coatings composition (11–17 at.%) (figure 3), what is apparently connected with maximal hardness values and minimal residual stress for the given concentration range.



1 – TiN, 2 – (Ti,Zr)N (7 at. % Zr), 3 – (Ti,Zr)N (11 at. % Zr),
4 – (Ti,Zr)N (17 at. % Zr), 5 – (Ti,Zr)N (21 at. % Zr)

Figure 3. The friction factor of the vacuum arc coated cutting plates.

The analysis of the scratches of the multicomponent (Ti,Zr)N coatings is evidence of that cracks and chips are appeared under the load P = 43,1 N, and it is confirmed by the amplitude rise of the acoustic emission and friction factor (figure 4).

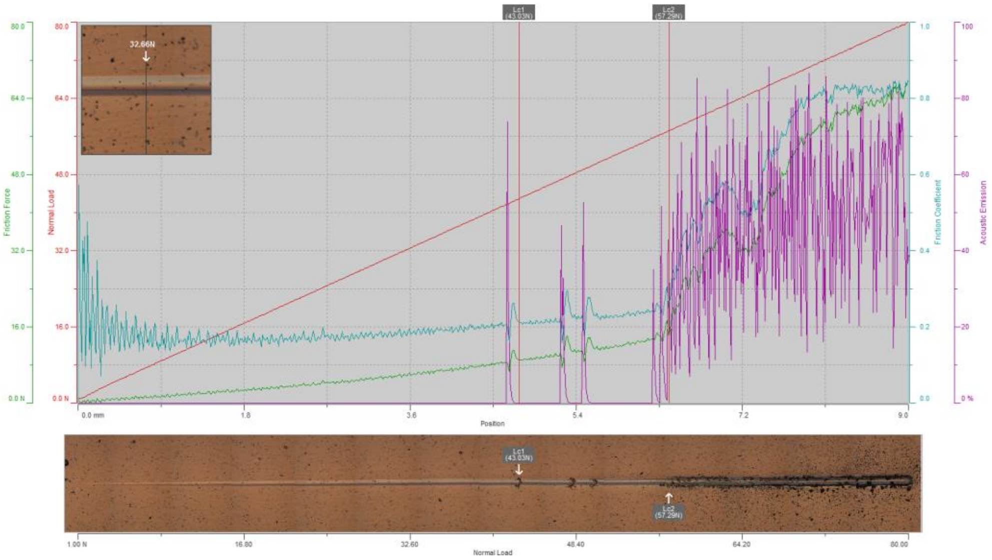
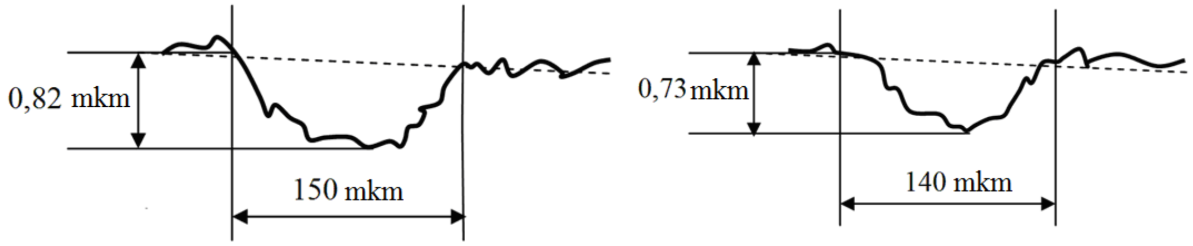


Figure 4. Scratch testing results of the multicomponent (Ti,Zr)N (17 at.%) coatings.

By means of scratch-testing is found, that the coatings are not peeled off under the scratching but are abrased, so they are destroyed by cohesion mechanism connected with plastic deformation and fatigue cracks generation in the coating material. The adhesion strength of the multicomponent (Ti,Zr)N coatings is more than 2,5 times in comparison with TiN (P = 16,13 N) [6].

The specific volume calculation of the coatings of the different composition was carried out by means of the friction track profilography results (figure 5).



a) b)

Figure 5. Friction track profilograms of (Ti,Zr)N coatings with different zirconium concentration: a) – (Ti,Zr)N (11 at. % Zr); b) – (Ti,Zr)N (17 at. % Zr).

As it should be from the wear diagrams, the multicomponent (Ti,Zr)N coatings, deposited from the separated plasma flows provides the substrate surface wear decreasing more than 3,5 times (figure 6).

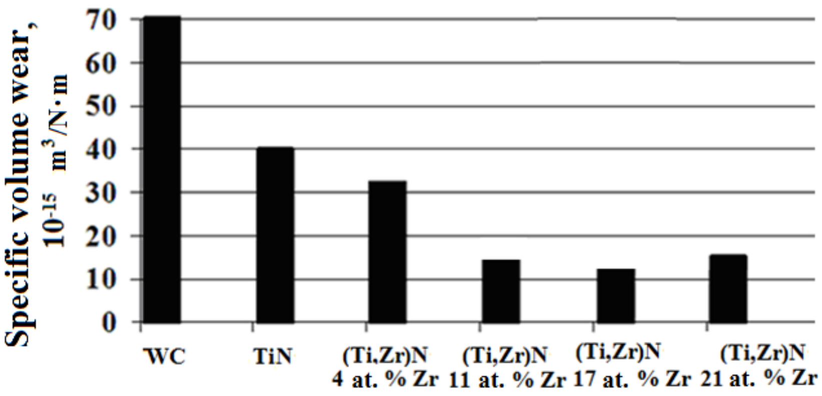


Figure 6. Specific volume wear of (Ti,Zr)N arc coated cutting plates with different zirconium concentration.

Besides, the multicomponent (Ti,Zr)N coatings with zirconium concentration in the coatings composition (11–17 at. %) has the best wear resistance.

CONCLUSIONS

The way of generation of the multicomponent (Ti,Zr)N coatings from the separated plasma flows, characterized by microcell structure and uniform elements distribution into depth is suggested.

The influence of the alloying element (Zr) on the structural characteristics and physical and mechanical properties of the multicomponent coatings is found.

It's determined, that microhardness of the multicomponent coatings (Ti,Zr)N increases up to 37 GPa, friction factor decreases to 0,45 and specific volume wear is 3,8 times lower in comparison with TiN coatings under the optimal technological deposition parameters.

REFERENCES

[1] P.H. Mayrhofer, Microstructural design of hard coatings / P.H. Mayrhofer, C. Mitterer, L. Hultman, H. Clemens // Progress in Materials Science. – 2006. – Vol. 51. – № 8. – P. 1032–1114.

[2] S.D. Latushkina, I.M. Romanov, O.I. Posylkina, V.M. Komarovskaya, O.Yu. Piskunova. Formation of wear-resistant nanostructured TiN/Cu coatings. – Journal of Friction and Wear, 2016. – V.37(1) – pp. 27–31.

[3] A.V. Chichinadze, Basis of tribology: manual for technical institutes / A.V. Chichinadze– M.: Machine-building, 2001. – 664 p.

[4] I.I. Aksenov, A.N. Belokhvostikov, V.G. Padalka, N.S. Repalov, V.M. Khoroshikh, Plasma flux motion in a toroidal plasma guide // Plasma Physics and Controlled Fusion. – 1986.– Vol. 28, υ 5.– P. 761–770.

[5] Nanohardness and Residual Stress in TiN Coatings / L.C. Hernández [et al.]. – Materials, 2011. – Vol. 4. – P. 929–940.