

Study of nanostructured (Ti-Zr-Nb)N coatings' physical- mechanical properties obtained by vacuum arc evaporation

This content has been downloaded from IOPscience. Please scroll down to see the full text.

2016 IOP Conf. Ser.: Mater. Sci. Eng. 110 012031

(<http://iopscience.iop.org/1757-899X/110/1/012031>)

View [the table of contents for this issue](#), or go to the [journal homepage](#) for more

Download details:

IP Address: 150.254.110.12

This content was downloaded on 04/11/2016 at 09:55

Please note that [terms and conditions apply](#).

You may also be interested in:

[Resonance scattering of light in nanostructured metallic and ferromagnetic films](#)

Vladislav V Kurin

[Development and characterization of cobalt based nanostructured super hydrophobic coating](#)

H. Mohsin, U. Sultan, Y. F. Joya et al.

[Multifunctional nanostructured films](#)

Evgenii A Levashov and Dmitry V Shtansky

[Synthesis of nanostructured materials based on YBa₂Cu₃O_{7-y} and BiFeO₃](#)

D K Palchaev, Zh Kh Murlieva, M H Rabadanov et al.

[Gradient nanostructured surface of a Cu plate processed by incremental frictional sliding](#)

C Hong, X Huang and N Hansen

[Chiralization and ferroelectric state induction in nanostructured liquid crystals](#)

B Katranchev, M Petrov, P M Rafailov et al.

[Structural features of the hydride phase formation in nanostructured \$\alpha\$ -titanium](#)

Yu R Kolobov, V I Torganchuk, V N Fokin et al.

Study of nanostructured (Ti-Zr-Nb)N coatings' physical-mechanical properties obtained by vacuum arc evaporation

S V Plotnikov¹, A D Pogrebnjak², L N Yerokhina¹, D K Yeskermessov¹, N K Yerdybaeva¹

¹*Ust-Kamenogorsk, 070003, Kazakhstan, D. Serikbayev East Kazakhstan State Technical University, 23/2-54 Serikbayev Street*

²*Sumy, 40000, Ukraine, Sumy State University, Rimsky-Korsakov Street, 2*

E-mail: didar.eskermesov@mail.ru

Abstract.

The coatings were formed by vacuum arc deposition. Unit cast target (cathodes) was used on the basis of 30 atm. % Ti, 35 at. % Zr and 35 atm. % Nb as the vaporized materials. Molecular nitrogen was used as the working gas. The thickness of the coatings in the experiments was 4.0 microns. The surface morphology fractograph fracture, track friction were investigated in a scanning electron microscope JSM-6390 LV. The use of multicoatings based on carbides is very promising to ensure the high performance properties of the complex, nitrides and silicides of transition metals.

Findings – nanostructured coating of (Ti-Zr-Nb) N was obtained by vacuum arc evaporation cathode-cast in a nitrogen gas reaction medium. Multicomponent films have a pronounced columnar structure. Elemental composition was obtained by the vacuum arc deposition of coatings (Ti-Zr-Nb) N, depending on the physical parameters of the deposition process, in particular the pressure of the reaction gas nitrogen.

1. Introduction

During the process of work the surface layer of machine elements and mechanisms are subjected to strong mechanical, heat and chemical influence. The loss of working functionality in most cases is the result of surface wear, erosion, corrosion, etc. Significant resource to increase the working operating capacity may be referred to material of which the details having been made. Due to this reason modern machine building pays great attention to tribotechnical material technology. To apply for this reason volumetric alloyed steel is often uneconomic and sometimes technologically non-profitable. However, the necessary results may be received by many functional coatings on working surfaces. They may simultaneously unite high hardness, wear resistance and heat endurance. For ensuring the complex of high working service property the usage of multielements coatings on the bases of carbides, borides, nitrides and silicides of transitional metals is rather perspective [1 – 4]. Stability and structure of composition, also high running quality of multicomponental elements of nitride systems make possible development of surface physic-mechanical characteristics and their application in the form of safety films protecting from ingress of contamination into subsurface of items layers [5, 6].

At present the most widely used are ion-plasmas techniques by film deposition coatings, in particular vacuum-arc and magnetron sputtering [7, 8].

We investigate the peculiarities of ion-plasma coatings by multielements system sputtering on the basis of Ti+Zr+Nb and we also perform analysis of physic-mechanical properties of the received coatings.



2. Experimental details

The coatings have been made by vacuum-arc depositions. For vaporizing materials we use solid accumulation electrode (cathode) on the basis of system: 30 at. % Ti, 35 at.% Zr and 35 at.% Nb. For active gas we apply molecular nitrogen. The thickness of all coatings in our experiments was 4.0 μm . Deposition parameters are given in Table 1.

Table 1. Physic-technological deposition parameters of coatings on the basis of (Ti-Zr-Nb)N.

# series	Vaporized material	I_o, A	U_{sm}, B	P_N, Torr
1a	Ti+Zr+Nb	95	100	3×10^{-4}
1b	Ti+Zr+Nb	95	100	7×10^{-4}
1c	Ti+Zr+Nb	95	100	4×10^{-3}

Surface morphology of fractography break, friction tracks were under research on scanning electron microscope JSM-6390LV. The research of elements composition of coatings was conducted by methods of X-ray characteristic spectrum analysis generated by electron beam in scanning electron microscope. X-ray structure research of samples with coatings were conducted on diffractometer DRON-4 in Cu- k_α radiation. The coatings hardness was determined with the help of hardness testing machine mode DM 8 according to micro-Vickers method with load on indenter 0.05 H. Adhesion-cohesion solidity, firmness to tracking and mechanism of coatings damages were under research in the air with the usage of scratch-tester Revetest (CSM Instruments). Tribological tests were conducted in the air according to scheme «ball-disk». As for friction machine we use «Tribometer», CSM Instruments. The coatings thickness comprised $\sim 3.5 - 4.0 \mu\text{m}$. For rider we use a ball 6.0 mm in diameter, made of sintered certified body - Al₂O₃. The load was 3.0 H, slide rate 10 sm/s. Tests correspond to international standards ASTM G99-959, DIN50324 and ISO 20808. The quantitative evaluation of samples wear resistance and rider were conducted according to wear-out factor W [9], methodology of calculation was given in work [10].

3. Results and discussion

Image of surface coatings and also fractography break are shown on Fig.1.

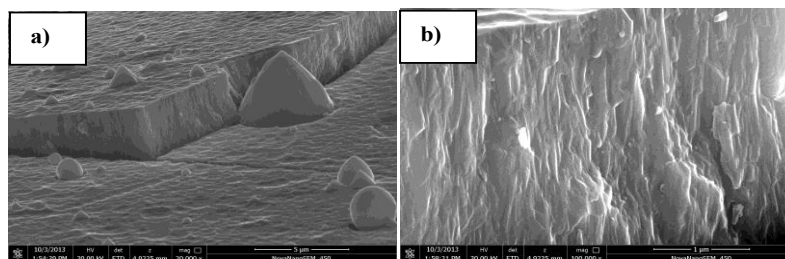


Figure 1. Image of coatings fractography break (Ti-Zr-Nb)N, received at nitrogen partial pressure: a – $P = 4 \times 10^{-3}$ Torr.; b – magnified image of coating part $P = 4 \times 10^{-3}$ Torr.

The research of surface morphology points that the pressure increase by nitric agent reaction leads to lessening of macroparticles quality and diameter.

That is particularly significant in active gases vacuum chamber forming with evaporated material refractory compounds [11]. We also observe the coating roughness lowering.

Coating element composition received by vacuum-arc deposition method was analyzed by energy dispersed method. (Table 2).

Table 2. Chemical composition of elements in coating (Ti-Zr-Nb)N.

# series	Composition of elements, at. %			
	N	Ti	Zr	Nb
<i>a</i>	38.72	20.91	20.38	19.99
<i>b</i>	40.00	22.57	18.04	19.39
<i>c</i>	40.86	20.52	19.36	19.26

If to compare elements composition of coatings series *b* and *c*, we can see that the first series samples the number of nitrogen atoms practically is equal. For samples *c* series, received at more higher pressure of N_2 atmosphere, the significant increase of zirconium atoms is noticeable, also several decrease of titanium atoms part. While this the niobium atoms for both series samples is actually the same. The increase of titanium atoms content in condensate of *b* series is possibly explained by more effective interaction of titanium atoms with nitrogen in subsurface layers.

The research of surface coatings fractography break (Fig. 1), received at different nitrogen partial pressure, testifies the formation of columnar structure (Fig. 1b), characteristic for coatings received by vacuum-arch precipitation method.

X-ray diffractometrical spectrum analysis presented on Fig.2 shows that the determining phase composition is phase with face-centered cubic alloy with crystal lattice. Low-intensity peak in section $2\theta = 38^\circ$ testifies the minor inclusion presence with BCC lattice characteristic while vacuum-arch method for drip-feed stage [12].

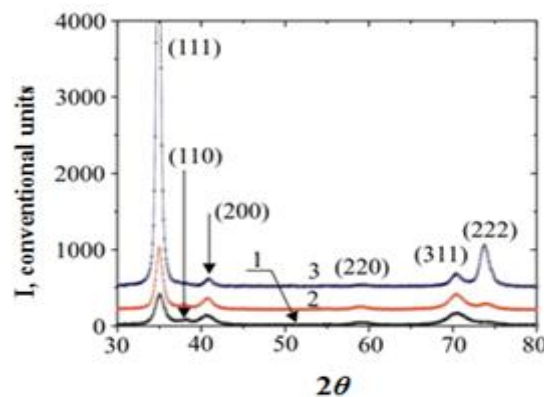


Figure 2. Sections of X-ray diffraction spectrums coatings received at different nitrogen partial pressures: curve 1 – $P = 3 \times 10^{-4}$ Torr; 2 – $P = 7 \times 10^{-4}$ Torr; 3 – $P = 4 \times 10^{-3}$ Torr; indicated planes of FCC lattice.

It should be marked that with pressure increase the peak intensity becomes lower (ibid. spectrum 1 and 3 on Fig. 2), it may be determined by significant decrease of drip-feed phase in coating and correlate with results of surface research. The set of planes $\{111\}$ increase is a characteristic feature that is determined by perfection increase of preferred crystal growth orientation with axis $[111]$ perpendicular to surface flatness.

The crystal size determined by approximation method with increase of pressure grows from 10 nm at lowest pressure 3×10^{-4} Torr up to 63 nm at highest nitrogen work pressure 4×10^{-3} Torr.

Adhesive-corrosion solidity research, endurance to coating tracking are given on Figures 3 – 4. Based on friction coefficient modification graph and acoustics emission from scribing load we determine the following critical loads: L_{C1} – the appearance of first chevron crack on the bottom and diagonal on the sides of tracking; L_{C2} – chevron cracks lots formation at the bottom of scratch and local chalking of coating, formation of chevron cracks at the bottom of scratch; L_{C3} – cohesive adhesive coating demolishing; L_{C4} – plastic abrasion of coating.

For criteria of adhesive firmness we adopt the critical load L_{C4} , leading to coating abrasion. Fig. 3 presents the dependence of friction coefficient and acoustic emission signal from applied load while *c* test samples scratch testing.

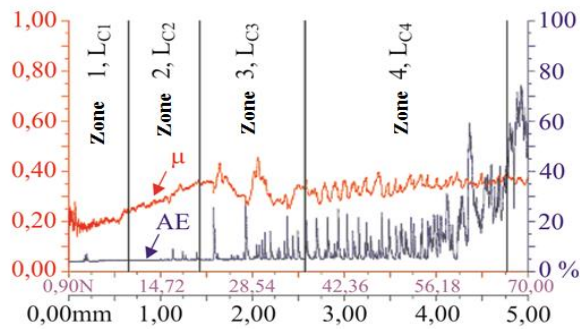


Figure 3. Dependence of friction coefficient from applied load while coating (Ti-Zr-Nb)N scratch testing received at $P = 4 \times 10^{-3}$ Torr.

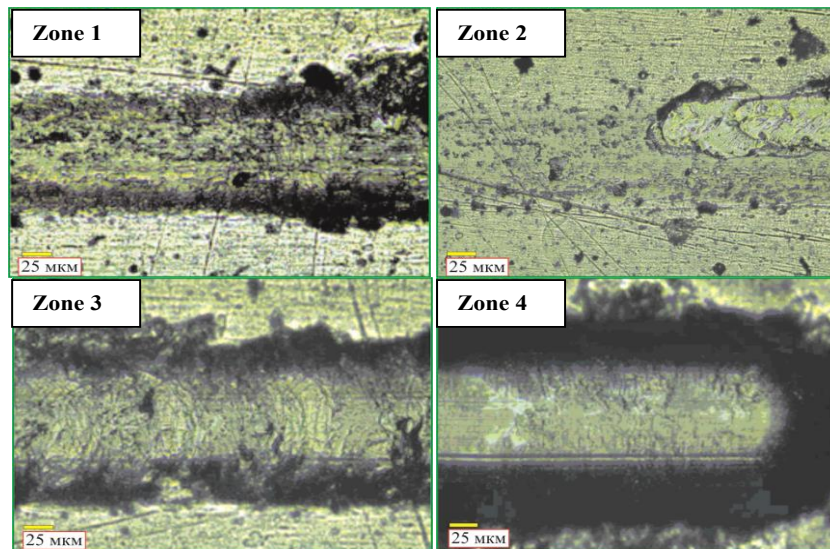


Figure 4. Diamond indenter contact zones with (Ti-Zr-Nb)N coating.

Conventionally the process of coating demolishing while indenter tracking may be divided into four stages. In the load range from $F = 0,9 H$ up to $F = 9.89 H$ indenter monotonous penetration comes into coating: friction coefficient slightly increases, acoustic emission signal preserved without changes At load $F = 15.81 H$ indenter fully submerges into coating.. The sliding of diamond indenter along the coating has friction coefficient 0.35.

According to load increase ($F = (20.6 - 36.4) H$) runs out of stuff occurs in front of the indenter in the form of knob and increases the depth of indenter penetration.

Table 3 shows the samples test results with coatings (Zr-Ti-Nb)N comparing with coatings (Ti-Zr-Si)N and TiN received by us [13].

Table 3. Comparative results of adhesive coatings tests in systems (Zr-Ti-Nb)N and (Ti-Zr-Si)N, TiN.

Critical loads	Coatings				TiN [12]
	(Zr-Ti-Nb)N	(Zr-Ti-Nb)N	(Zr-Ti-Nb)N	(Ti-Zr-Si)N	
	Series, a	Series, b	Series, c	[12]	
L_{C1}	2.91	0.9	9.89	3.91	21.31
L_{C2}	29.04	15.82	20.62	18.15	30.91
L_{C3}	43.18	42.37	36.43	24.29	40.28
L_{C4}	59.26	66.24	66.77	43.15	48.84

According to research [14] while testing for adhesive solidity depending upon different meanings of critical loadings several physic-chemical processes are being carried out simultaneously while abrasion, however only L_C is directly connected with adhesive demolishing.

The results of mechanical characteristics measurements, in particular hardness, received for coatings (Zr-Ti-Nb)N are given in Table 4.

Table 4. Average values of coatings hardness on the system (Zr-Ti-Nb)N base.

#series	hardness HV _{0.05} GPa	Endnote
<i>a</i>	37.21	Forward flow
<i>b</i>	40.21	Forward flow
<i>c</i>	44.57	Forward flow

The way it may be seen from Table 4 maximum meaning of hardness $H = 44.57$ GPa was received at pressure of reactionary gas $P = 4 \times 10^{-3}$ Torr may be referred according classification [15], to superhard coatings with hardness $HV_{0.05} = 40$ GPa. Before the wearing tests profile record of steel disks surfaces on which coverings were overcoated were taken off.

Overcoatings of (Zr-Ti-Nb)N system on steel disk polished surface leads to roughness increase at the account of drip-feed component of plasma flow.

The average meaning of friction coefficient (μ) in system «covering–Al₂O₃» while tests comprises 1.1.

As it follows from Table 6, coverings of system (Zr-Ti-Nb)N, received at reaction gas pressure $P = 4 \times 10^{-3}$ Torr, possess hard constituency.

Tribological tests of samples with coatings are given in Table 5. Under the friction conditions being held in our experiments, sample with covering is in more heavy conditions of loading comparing with rider. Rider's surface – ball is under constant loading and constantly undergoes cyclic loading connected only with elastic joining, breaking of adhesion tires in the limits of separate micro areas of real contact one and the same area

Table 5. Tribological characteristics of covering system «covering (Zr-Ti-Nb)N – Al₂O₃».

#series	Friction coefficient, μ		Wear factor, $\text{mm}^3 \times \text{H}^{-1} \times \text{m}^{-1}$	
	Initial	During tests	Rider ($\times 10^{-5}$)	Sample ($\times 10^{-5}$)
<i>a</i>	0.61	1.95	0.391	9.69
<i>b</i>	0.45	1.19	2.84	3.1
<i>c</i>	0.491	1.05	3.21	2.4

The results of friction tracks elements' analysis are given in Table 6.

Table 6. Friction tracks of elements composition.

#series of samples	elements, at. %							
	N	O	Al	Zr	Nb	Ti	Fe	Mn
<i>a</i>	41.5	11.58	0.28	11.86	12.57	22.54	-	-
<i>b</i>	43.66	11.79	0.32	11.12	11.42	21.24	0.34	-
<i>c</i>	-	41.98	0.64	1.7	1.73	3.32	50.37	0.27

It is well known that while forecasting the working characteristic of different wares connected with friction it is necessary to treat friction characteristics of tribosystem (body - rider) not for separately taken element but in general because structural-phase coating condition and its physic-mechanical characteristics plays decisive role in the process of tribosystem operation.

4. Conclusion

1. We received nanostructural coating systems (Ti-Zr-Nb)N by method of solid cathode vacuum-arc evaporation in the medium of reacting gas nitrogen Multicomponent films have clearly expressed columnar structure.

2. X-ray analysis displays that the basic phase is the phase with face-centered cubic crystal lattice. Nanocrystals dimensions increase under pressure up to 10 nm from minor pressure of 3×10^{-4} Torr up to 63 nm while the major pressure of running nitrogen atmosphere is 4×10^{-3} Torr. While pressure increase of reacting gas we observe set of peaks reinforcement {111}, that may be determined via the perfection of crystal growth increase with primary axial orientation [111] perpendicular to plane of growth.

3. Physic-technological parameters on solid coverings deposition were explored. Covering system (Ti-Zr-Nb)N hardness received at partial pressure $P = 4 \times 10^{-3}$ Torr forms $H_{0.05} = 44.57$ GPa, and at pressure $P = 3 \times 10^{-3}$ Torr hardness makes $H_{0.05} = 37.21$ GPa.

4. For covering series 1c on the system basis (Ti-Zr-Nb)N, overlaid on steel X18H10T substrate material, the starting point of cracks L_{C2} appearance occurs at load $F = 20.62$ GPa, adhesive destruction at load $F = 66.77$ GPa. In case 1c series L_{C2} occurs at $F = 29.14$ GPa, and destruction appears at $F = 59.26$ GPa, that is 15 % higher.

5. Adhesion solidity of coverings on the basis (Ti-Zr-Nb)N is evidently higher comparing with coverings on the basis (Ti-Zr-Si)N and TiN, and adhesion destruction is observed at load $F = 66.77$ GPa for covering (Ti-Zr-Nb)N for covering on the basis (Ti-Zr-Si)N $F = 48.84$ GPa; and for TiN – $F = 55.2$ GPa.

6. Coverings wear resistance on the system basis (Zr-Ti-Nb)N series 1c is higher than wearing resistance of covering on basis (Zr-Ti-Nb)N series 1a. Significance of friction coefficient for coating (Zr-Ti-Nb)N while system testing «covering – Al_2O_3 » makes $\mu \sim 1.1$.

References

- [1] Pogrebnjak A D, Shpak A P, Azarenkov N A, Beresnev B M. 2009 *Struktura I svoistva tverdih I sverhtverdi nanokompozitnih pokritii.UFNA*. Vol. 1 pp. 35-64.
- [2] Pogrebnjak A D. 2013 Structure and Properties of Nanostructured (Ti-Hf-Zr-V-Nb)N Coatings. *J. of Nanomaterials*. Article ID 780125, pp. 12.
- [3] Azarenko N A, Sobol O V, Beresnev B M, Pogrebnjak A D, Kolesnikov D A, Turbin P V, Toryanik I N. 2013 Vakuumno-plazmennie pokritii na osnove mnogoelementnih nitridov. *Metallofizika noveishie tehnologii*. Vol. 8 pp. 1001-1024.
- [4] Pogrebnjak A D, Beresnev V M, Demianenko A A, Baidak V S. 2012 Adhesive strength, superhardness, and the phase and elemental compositions of nanostructured coatings based on Ti-Hf-Si-N. *Physics of the Solid State*. Vol. 54 pp. 1882-1890.
- [5] Veprek S, Veprek-Hejman M G L, Karvankova P, Prohazka J. 2005 Different approaches to superhard coatings and nanocomposites. *Thin Solid Films*. Vol. 476 pp. 1-29.
- [6] Musil J, Baroch P, Zeman P. 2008 Hard nanocomposite coatings. Present Status and Trends: in Book Edit. R Wei “*Plasma Surface Engineering Research And Its Practical Applications*”. Kerala Research Signpost Publ, pp. 1-34.
- [7] Andreev A A, Sablev L P, Grigorev S N. 2010 *Vakuumno-dugovie pokritiya*. X.: NNC HFTI, pp. 317.
- [8] Berlin E V, Seidman L A. 2010 Ionno-plazmennie processi v tonkoplennchatoi tehnologii. *M.: Tehnosfera*, pp. 528.
- [9] Ibatullin I D. 2008 *Kinetika ustalostnoi povrezhdaemosti I razrusheniya poverhnostnih sloev: monographiya*. Samara: Samarskii gosudarstvenni tehnikeskii universitet, pp. 396.
- [10] Verzhinin D S, Smolyakova M U, Manohin S S, Druchinina O A, Ahmadeev U H. 2010 Issledovanie tribologicheskikh svoistv azotirovannogo titanovogo splava BT16 s ispolzovaniem avtomatizirovannoi mashini treniya. *Zavodskaya laboratoriya. Diagnostika materialov*. Vol. 12 pp. 45-49.
- [11] Aksenov I I, Andreev A A, Belous B A, Strelnickii B E, Horoshih B M. 2012 *Vakuumnaya duga: istochniki plazmi, osazhdenie pokritii, poverhnostnoe modifitsirovanie*. K.: Naukova dumka, pp. 727.
- [12] Azarenkov N A, Sobol O V, Beresnev B M, Pogrebnjak A D, Litovchenko S V, Ivanov O N. 2013 *Materbalovedenie neravnovesnogo sostoyaniya poverhnosti*. Summi: SumGU, pp. 683.
- [13] Pogrebnjak A D, Yakushchenko I V, Bagdasaryan A A, Bondar O V, Krause-Rehberg R, Abadias G, Chertier P, Oyoshi K, Takeda Y, Beresnev V M, Sobol O V. 2014 Mycrostructure, Physical and Chemical Properties of nanostructured (Ti-Hf-Zr-V-Nb)N coatings under different deposition conditions. *Mater. Chem. and Phys*. Vol. 147 pp. 1079-1091.
- [14] Valli J. 1986 A review of adhesion test methods for thin hard coatings. *Journal of Vacuum Science and Technology*. Vol. 4 pp. 3007-3014.
- [15] Azarenkov N A, Beresnev B M, Pogrebnjak A D, Kolesnikov D A. 2013 Nanostrukturnie pokritiya I nanomateriali. *Osnovi polucheniya, svoistva, oblasti primeneniya*. M.: Knijni dom «Librikom», pp. 368.