

## Physical and Mechanical Properties of (Ti-Zr-Nb)N Coatings, Fabricated by Vacuum-Arc Deposition

O.V. Bondar<sup>1</sup>, K.O. Belovol<sup>1</sup>, O.V. Maksakova<sup>1,\*</sup>, V.M. Beresnev<sup>2</sup>, O.V. Sobol<sup>3</sup>, S.S. Grankin<sup>2</sup>,  
 U.S. Nyemchenko<sup>2</sup>, V.Yu. Novikov<sup>4</sup>, D.K. Eskermesov<sup>1,5</sup>

<sup>1</sup> Sumy State University, 2, Rymsky Korsakov Str., 40007 Sumy, Ukraine

<sup>2</sup> V.N. Karazin Kharkiv National University, 4, Svobody Sq., 61022, Kharkiv, Ukraine

<sup>3</sup> National Technical University "Kharkiv polytechnic institute", 21, Frunze Str., Kharkiv 61002, Ukraine

<sup>4</sup> National Research University "Belgorod State University", 85, Pobedy Str., Belgorod, 308015, Russia

<sup>5</sup> D. Serikbaev East Kazakhstan State Technical University,  
 69, Protozanov Str. Ust-Kamenogorsk 070004, Kazakhstan

(Received 10 August 2015; published online 26 August 2015)

The coatings based on (Ti-Zr-Nb)N were fabricated by vacuum-arc deposition of the Ti+Zr+Nb cathodes in the nitrogen atmosphere. Their physical and mechanical properties as well as tribological characteristics have been studied. The coatings are characterized by a columnar structure, their hardness reaches 44.57 GPa. The adhesion strength of coatings reaches 66.77 GPa, the friction coefficient of the «cover – Al<sub>2</sub>O<sub>3</sub>» is 1.1. It has been determined, that the hardness of the investigated coatings significantly depends on the pressure of the reaction gas. The coatings are promising as protective coatings for friction pairs and cutting tools.

**Keywords:** Superhard Coatings, Structure, Wear, Adhesion, Nanostructure.

PACS numbers: 81.15.Ef, 81.15.-z

### 1. INTRODUCTION

To provide complex high performance properties very promising can be the use of multi coatings based on carbides, borides, nitrides and silicides of transition metals [1–3]. The stability of the structure and composition, as well as high performance multi-element nitride systems provide improvement of physical and mechanical characteristics of the surface and use of them as a protective film prevents the penetration of harmful impurities in the surface layers of articles [4, 5]. Currently, the most widely used are methods of ion-plasma deposition of coatings, in particular vacuum-arc and magnetron sputtering [6, 7].

In this work features of formation of ion-plasma coatings by spraying a multi-element systems based on Ti+Zr+Nb were investigated and an analysis of the physical and mechanical properties of the coatings was made.

### 2. DESCRIPTION OF NANOCOMPOSITE DEPOSITION AND INVESTIGATION

The coatings were formed by vacuum arc deposition. As the evaporating materials used solid target (cathodes) based on system: 30 at. % Ti, 35 at. % Zr and 35 at. % Nb. As working gas used molecular nitrogen. The thickness of all coatings in the experiments was 4.0 microns.

Investigation of the elemental composition of coatings was conducted by analyzing the spectra of the characteristic X-rays generated by an electron beam in raster electron microscope. The spectra were recorded using a X-ray energy dispersive spectrometer system PEGASUS company EDAX, installed in the microscope.

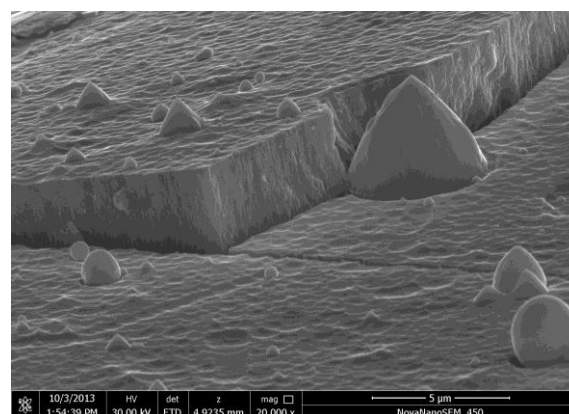
X-ray diffraction studies of the samples with coating was carried out on diffractometer DRON-4 Cu-K<sub>α</sub> radiation.

The microhardness measurements were carried out by using an automatic system for analysis of DM-8 microhardness with a load on the indenter 0.05 H by the micro-Vickers method.

Adhesion-cohesive strength, scratch resistance and failure mechanism of coatings was performed on the air using the scratch tester Revetest (CSM Instruments).

### 3. RESULTS AND DISCUSSION

Image surface of the coatings as well as fractographs fracture showed on Fig. 1.



**Fig. 1** – Images of fractographs fracture of the coatings (Ti-Zr-Nb)N, obtained at a partial pressure of nitrogen: P = 4×10<sup>-3</sup> Torr

\* [maksakova.tereshenko@gmail.com](mailto:maksakova.tereshenko@gmail.com)

The investigation of the surface morphology indicates that increasing of pressure of the reaction nitrogen environment reduces the amount and size of macroparts.

This is particularly important at presence in the vacuum chamber of reactive gases, forming with vaporizable material refractory compound [8]. Also, a decrease in the roughness of the coating.

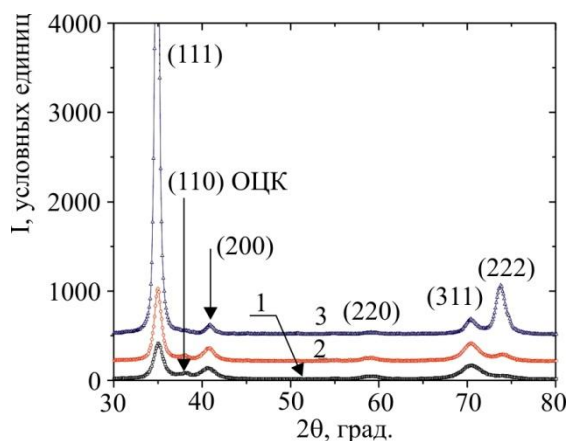
The elemental composition of coatings produced by vacuum arc deposition was analyzed by energy dispersive method (Table 1).

**Table 1** – Chemical composition of elements in the coating (Ti-Zr-Nb)N

Series No	Elemental composition, at. %			
	N	Ti	Zr	Nb
a	38,72	20,91	20,38	19,99
b	40,00	22,57	18,04	19,39
c	40,86	20,52	19,36	19,26

The analysis X-ray diffractometer spectra on Fig. 2 shows that as the determining phase composition is the phase with a face-centered cubic lattice. Low-intensity peak at  $2\theta = 38^\circ$  indicates the presence of small inclusions with BCC lattice, typical for vacuum-arc method for dropping phase [9].

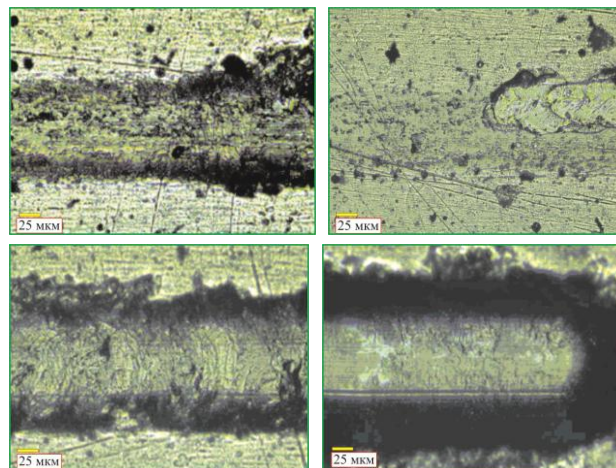
It should be noted that with increasing of pressure the intensity of this peak decreases (see. the spectra 1 and 3 on Fig. 2), which determined by a significant decrease of content in the droplet phase in the coating and correlates with the results of surface examination.



**Fig. 2** – Areas of the diffraction spectra of coatings obtained at different partial pressure of nitrogen: curve 1 –  $P = 3 \times 10^{-4}$  Torr; 2 –  $P = 7 \cdot 10^{-4}$  Torr; 3 –  $P = 4 \times 10^{-3}$  Torr; identified planes of FCC lattice

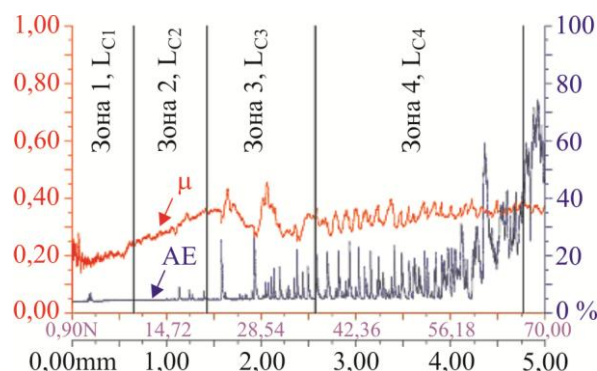
The study results of the adhesive-cohesive strength, scratch resistance of coatings shown on Fig. 3 and Fig. 4. On the basis of the graphs the change of the friction coefficient and acoustic emission from the load of scribing determined the following main critical loads:  $LC1$  – the emergence of the first chevron cracks on the

bottom and diagonal around the edges of crack;  $LC2$  – the formation of a plurality of chevron cracks on the bottom of the crack and local peeling of coating, appearing of chevron cracks on the bottom of the crack;  $LC3$  – cohesively-adhesion failure of the coating;  $LC4$  – plastic abrasion of the coating.



**Fig. 4** – Zones of contact of diamond indenter with the coating (Ti-Zr-Nb)N

As criteria of the adhesion strength was accepted critical load  $LC4$ , leading to abrasion of the coatings. Fig. 3 shows the dependence of the friction coefficient and acoustic emission signal from the applied load at scratch test samples of series b.



**Fig. 3** – Dependence of friction coefficient on the applied load at a scratch test of the coating (Ti-Zr-Nb)N, obtained at  $P = 4 \times 10^{-3}$  Torr

Conventionally, the process of destruction of the coating in scratching with the indenter can be divided into four stages. At the load range of  $F = 0.9$  H to  $F = 9.89$  H appear the monotonically penetration of the indenter into the coating: the friction coefficient slightly increases the acoustic emission signal remains unchanged. When the load  $F = 15.81$  H indenter is completely immersed in the coating. Slipping diamond indenter for cover runs with friction coefficient 0.35.

As the load increases ( $F = (20.6 - 36.4)$  H) occurs the extrusion of the material before the indenter as hillocks and increased the penetration depth of the indenter.

#### 4. CONCLUSIONS

With method of vacuum arc evaporation solid cathode in the medium of reaction gas nitrogen obtained nanostructured coating of (Ti-Zr-Nb)N. Multicomponents films have a pronounced columnar structure.

Elemental composition of obtained with vacuum-arc deposition coatings (Ti-Zr-Nb)N, depends on the physico-technological parameters, by precipitation, particularly on the pressure of the reaction gas nitrogen.

From X-ray analysis follows that the main phase is a face-centered cubic lattice. Dimensions nanocrystals with increasing of the pressure increases from 10 nm at the lowest pressure of  $3 \times 10^{-4}$  Torr to 63 nm at the maximum working pressure nitrogen atmosphere  $4 \times 10^{-3}$  Torr.

Investigation of the effect of physical and technological parameters of deposition on the hardness of coatings. The hardness of the coatings of (Ti-Zr-Nb)N system, obtained at a partial pressure of  $P = 4 \times 10^{-3}$  Torr is  $H_{0,05} = 44.57$  GPa, and at a pressure  $P = 3 \times 10^{-3}$  Torr hardness is  $H_{0,05} = 37.21$  GPa.

The adhesion strength of coatings based on (Ti-Zr-Nb)N markedly higher compared to coatings based on (Ti-Zr-Si)N and TiN, and the adhesion failure is observed at the load  $F = 66.77$  GPa for coating (Ti-Zr-Nb)N for coating based on (Ti-Zr-Si)N  $F = 48.84$  GPa; and for TiN  $F = 55.2$  GPa.

#### ACKNOWLEDGEMENTS

The work performed by the authors in the framework of complex state budget scientific research works number 0113U001079, 0112U005920 and 0112U006974, 41124001382, funded by the Ministry of Education and Science of Ukraine. The part of the work performed on the diagnostic equipment of the Center for collective use of scientific equipment of Belgorod National Research University "Diagnosis of the structure and properties of nanomaterials" the Ministry of Education and Science of the Russian Federation.

#### REFERENCES

1. A.D. Pogrebnjak, A.P. Shpak, N.A. Azarenkov, V.M. Beresnev, *Phys. Usp.* **1**, 179 (2009).
2. Alexandr D. Pogrebnjak, *J. Nanomater.* **2013**, 780125 (2013).
3. A.D. Pogrebnjak, V.M. Beresnev, A.A. Demianenko, V.S. Baidak, F.F. Komarov, M.V. Kaverin, N.A. Makhmudov, D.A. Kolesnikov, *Phys. Solid State.* **9**, 54 (2012).
4. S. Veprek, M.G.L. Veprek-Hejman, P. Karvankova, J. Prohazka, *Thin Solid Films* **476**, 1 (2005).
5. J. Musil, P. Baroch, P. Zeman, *Plasma Surface Engineering Research And Its Practical Applications.* (Kerala Research Signpost Publ.: 2008).
6. *Vacuum-arc Coatings* (A.A. Andreev, L.P. Sablev, S.N. Grigor'ev) (Kharkiv: NSC KhFTI: 2010).
7. *Ion-plasma Processes in Thinfilms Technology* (E.V. Berlin, L.A. Seydman) (Moscow: Technosphere: 2010).
8. *Vacuum-arc: Sources of Plasma, Coatings Deposition, Surface Modification* (I.I. Aksenov, A.A. Andreev, V.A. Belous, V.E. Strel'nitskyi, V.M. Khoroshyh) (Kyiv: Scientific Thought: 2012).
9. *Materialscience Nonequilibrium State of Modified Coating* (N.A. Azarenkov, O.V. Sobol', V.M. Beresnev, A.D. Pogrebnjak, S.V. Litovchenko, O.N. Ivanov) (Sumy: SumDU: 2013).