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Dependence of mechanical and tribotechnical properties of multilayered TiN/ZrN coatings on deposition

Abstract. *C-PVD method (vacuum arc deposition of a cathodes) was used for deposition of multilayered coatings, based on TiN/ZrN systems, with different thickness of bilayers. Total thickness of the coatings was 11 – 19 μm, thickness of bilayers varied from 39 to 305 nm for different samples depending on deposition conditions. Microstructure of the coatings, mechanical and tribotechnical properties, and wear resistance were explored. Influence of the bilayer thickness on such properties of the coatings, as hardness, elasticity modulus, wear resistance and wear ratio were explored.*

Streszczenie. *Metoda C-PVD (napylanie próżniowo-łukowe katod) została użyta do osadzania wielowarstwowych powłok, na bazie układów TiN/ZrN o zmiennej grubości pojedynczych warstw. Całkowita grubość powłoki wynosiła 11 – 19 μm, przy czym grubość pojedynczych warstw wahała się od 39 do 305 nm dla różnych próbek zależnie od warunków osadzania. Zbadano mikrostrukturę powłok, właściwości mechaniczne i trybologiczne oraz odporność na zużycie. (**Zależność właściwości mechanicznych i trybologicznych wielowarstwowych powłok TiN/ZrN od warunków osadzania**).*

Keywords: C-PVD method, multilayered, TiN/ZrN, bilayer, hardness, wear.
Słowa kluczowe: metoda C-PVD, wielowarstwowy, TiN/ZrN, dwuwarstwowy, twardość, zużycie.

Introduction

One of the possible ways to improve properties of the coatings based on nitrides and carbides of refractory metals is creation of the multilayered multifunctional coatings [1,2]. They can be widely used to increase lifetime of the high-speed cutting tools operating under the influence of high temperatures, as well as to increase the reliability of different friction pairs. A review of scientific sources [3-16], and our own experience in this area [17-19] showed, that multilayered coatings, which consist of alternating 20-30 nm layers of hard refractory metals nitrides, are rather perspective in modern materials science. Such coatings are characterized by high hardness, because the alternating stress fields prevent the movement of dislocations.

We should also mention, that multilayered, multicomponent and nanostructured coatings are widely used in modern materials science for increasing of protective properties of different industrial products, and for improving their hardness, wear and corrosion resistance, oxidation resistance under the influence of high temperatures and so on. That is why mechanical and tribotechnical characteristics of multilayered TiN/ZrN coatings with various thickness of nanolayers have been studied in the presented paper.

Experimental details

The multilayered coatings were fabricated using vacuum - arc deposition method [5,8,12] from two evaporators, one of which contained titanium of the grade VT1-00 (Fe < 0.12 %, C < 0.05 %, Si < 0.08 %, N < 0.04 %, O < 0.1, H < 0.008, Ti was in the range 99.5 – 99.9), the second – zirconium, which was fabricated using the method of electron beam melting using BULAT-6 deposition device, which allows deposition of nanostructured coatings in pulsed mode with variable pulse amplitude and pulse frequency [19]. Ion-plasma deposition or coating's deposition using vacuum arc cathode evaporation seems to be a very promising way of fabrication of protective coatings [20-32]. The coatings were deposited on the A 570 Grade 36 steel (R_a = 0.09 μm) polished substrates under different deposition regimes. Size of the substrates was 15x15x2.5 mm. We prepared three series of samples with different bilayer thickness and total thickness of the coatings (see Table 1). The arc current during deposition was 100 A, the nitrogen pressure in the deposition chamber was $3 \cdot 10^{-3}$ Torr, the distances from evaporators to substrate were 250 mm, the substrate temperature was 250…350°C, deposition speed of ZrN and TiN layers was around 3 nm/sec and 2 nm/sec respectively. Negative bias potential -30…-200 V was applied to the substrates. Such relatively low substrate temperature allowed us to fabricate homogeneous coatings with good enough planarity.

Table 1. Bilayer parameters and total thickness of the TiN/ZrN coatings

Series	Thickness of TiN interlayer [nm]	Thickness of ZrN interlayer [nm]	Bilayer thickness [nm]	Total thickness of the coating [μm]
1	19	20	39	19
2	35	36	71	11
3	81	124	305	13

Surface morphology, fracture patterns and friction tracks were studied using FEI Nova NanoSEM 450 scanning electron microscope. Structure-phase investigations were done on the DRON-3M diffractometer in Cu-Kα radiation. Tribological tests were done using CSM Instruments Tribometer in air by a "ball-disk" scheme. A 6 mm diameter ball, made of certified sintered material Al_2O_3, was used as a counterbody, load was 6 N, sliding speed was 10 cm/sec. Tests were done in according with international standards ASTM G99-959, DIN50324 and ISO 20808. Hardness of the coatings were measured by the method of micro-Vickers using DM-8 hardness tester. Load on the indenter was 0.2 N.

Results and discussion

Images of the cross-sections of the coatings of the Series 1 are presented in the Fig. 1. We can see that investigated coatings have rather good planarity of the deposited layers without droplet defects between TiN and

ZrN layers and inside them. The layers are clearly visible; they also have clear boundaries and do not intersect.

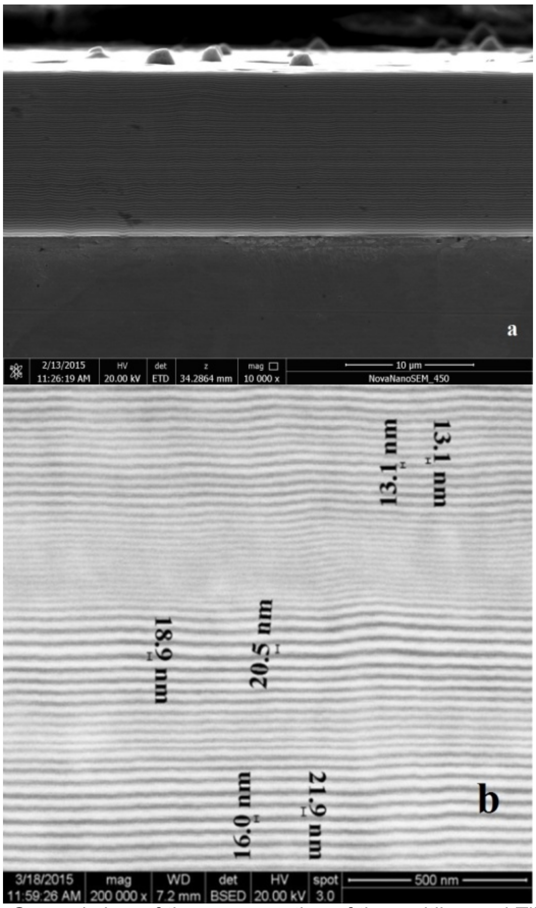

Fig.1. General view of the cross-section of the multilayered TiN/ZrN coating from the Series 1: magnification x10^4 (a) and x10^5 (b)

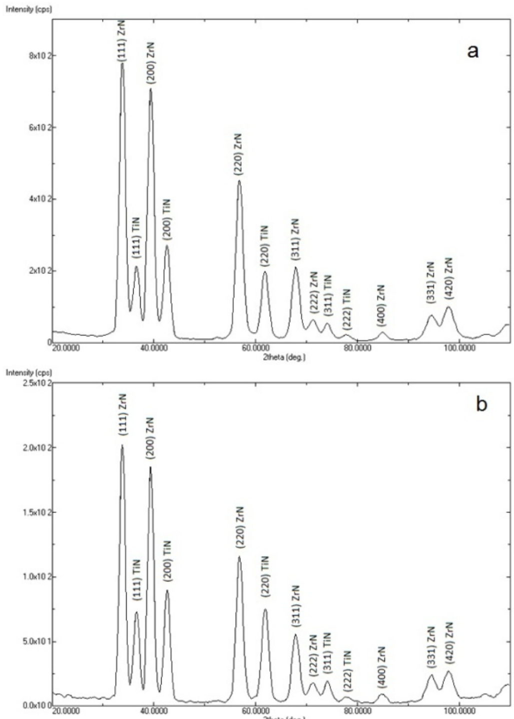

Fig.2. XRD spectra parts of the TiN/ZrN coatings from the Series 1 (a) and Series 2 (b)

According to X-ray diffraction investigations, we observe the formation of two-phase state of TiN and ZrN phases with a crystal lattice structure of the NaCl type with development of the preferred orientation with the [111] axis, perpendicular to the plane of growth, either in the TiN layers or in the ZrN layers. Results of XRD investigations of multilayered TiN/ZrN coatings with different multilayer thickness are presented in the Fig. 2. Planes are indicated above the respective peaks.

General surface views of the coating from Series 2 and 3 with different magnifications are presented in the Fig. 3. We can see, that the surface is rather smooth, and do not contains many droplets, that can influence significantly on the mechanical and tribotechnical properties of the coatings.

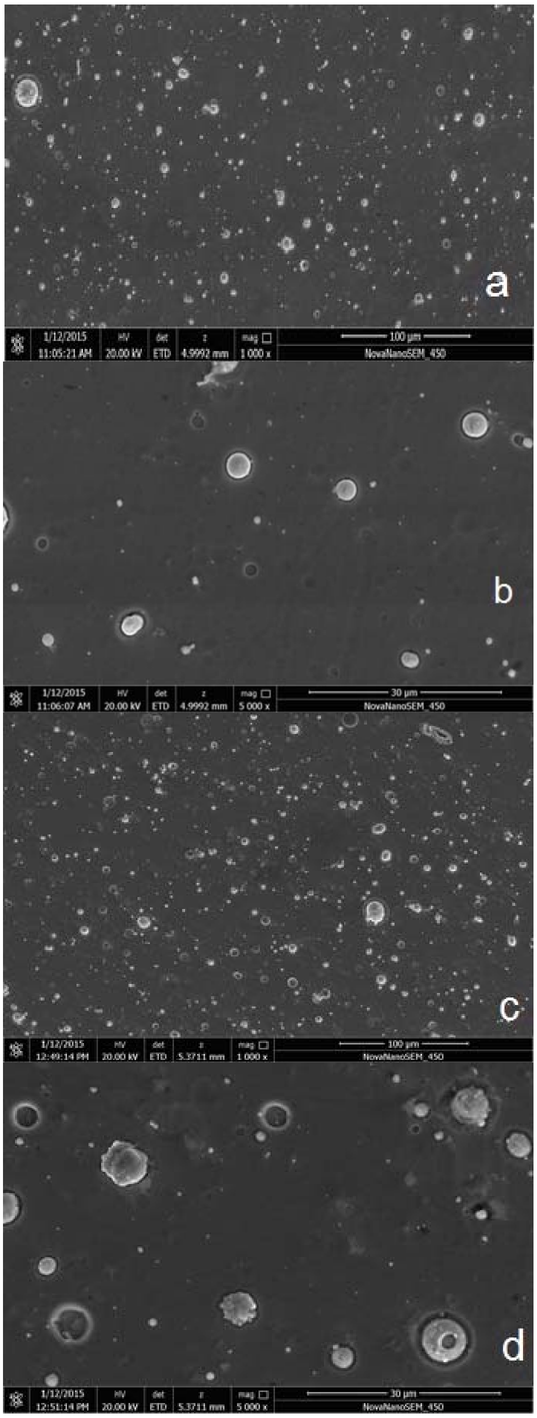

Fig.3. Surface view of the coatings from Series 2 (x10^3 – a, x5•10^3 – b) and Series 3 (x10^3 – c, x5•10^3 – d)

Fig. 4 shows the typical surface morphologies of the wear tracks. We obtained smooth wear track morphology with particle-like wear debris along the track slides. In addition, no fragmentation is observed inside and outside the track during the wear test. Track width was equal to 545.8…625.9 μm for samples from different series.

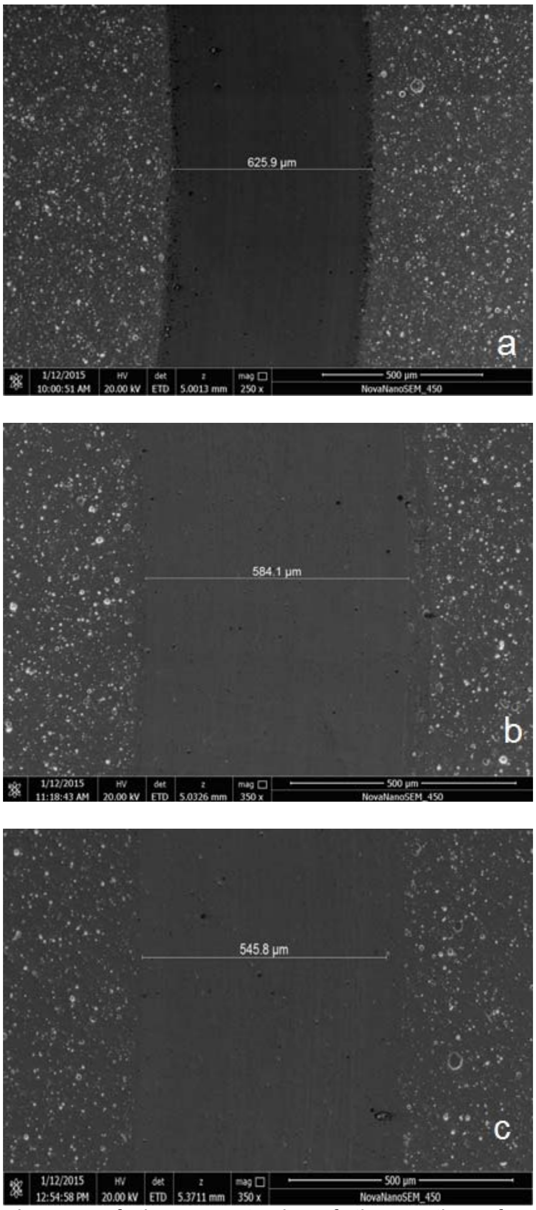

Fig.4. Images of the wear tracks of the coatings from the Series 1 (a), 2 (b) and 3 (c)

Generalized results of tribological investigations of the TiN/ZrN coatings with different ratios of bilayers are presented in the Table 2.

Table 2. Results of tribological investigations of the TiN/ZrN coatings

Bilayers ratio, TiN/ZrN [nm]	Friction coefficient		Wear factor, [$mm^3 \cdot N^{-1} \cdot mm^{-1}$]	
	Beginning	During tests	Counterbody ($x10^{-6}$)	Samples ($x10^{-5}$)
Series 1, 19/20	0.59	1.0	2.2	1.4
Series 2, 35/36	0.62	1.2	2.0	1.5
Series 3, 81/124	0.62	1.1	1.9	1.3

We can see, that fabricated coatings have high wear coefficient paired with the Al_2O_3 ball. All coatings have good wear resistance, the reduced wear was $(1.3 \div 1.5) \cdot 10^{-5}$ $mm^3 \cdot N^{-1} \cdot mm^{-1}$. Counterbody wear was also small $(1.9 \div 2.2) \cdot 10^{-5}$ $mm^3 \cdot N^{-1} \cdot mm^{-1}$. Splitting, cracking and peeling of the coatings were not observed. Good adhesion of the fabricated coatings to substrate were found. Coatings were plastically deformed during abrasion tests; observed wear was rather typical for soft metals [33]. Wear profile for the coating from the Series 3 is presented on the Fig. 5.

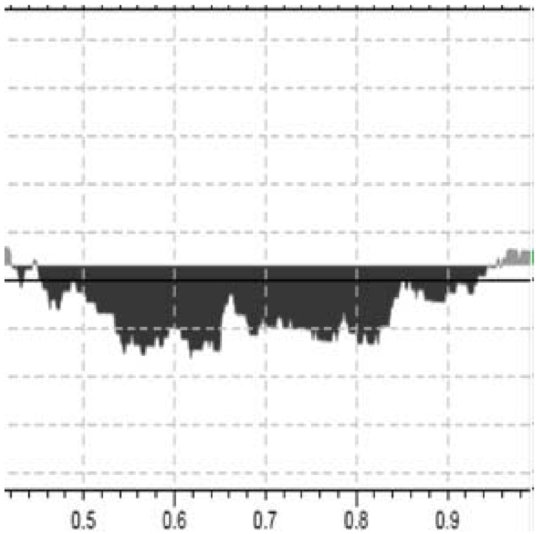

Fig.5. Wear profile of the coating from Series 3 after wear tests by the method of micro-Vickers

As it was mentioned above, the hardness of the coatings were measured by the method of micro-Vickers using DM-8 hardness tester. Based on the works [6, 8-10, 15-32] we assumed, that investigated coatings will perform high hardness in comparison with so-called monolayered coatings. Load on the indenter was 0.2 N. Average hardness of the coatings were 30.14 GPa for samples from the Series 1, 34.43 GPa for samples from the Series 2, 38.21 GPa for samples from the Series 3.

Conclusions

Multilayered TiN/ZrN coatings with different bilayer thickness were fabricated using vacuum-arc deposition method. Total thickness of the coatings were 11-18 μm, thickness of the bilayers was 39…305 nm. Formation of the two-phase state of TiN and ZrN phases with a crystal lattice structure of the NaCl type with the preferred orientation with the [111] axis, perpendicular to the plane of growth, was found in the coatings. Fabricated coatings demonstrated good wear resistance and adhesion to the substrate, as well as rather high hardness, which achieved the values of about 38 GPa, which is impossible for simple monolayered coatings. Thus, we can state, that the results of tribological tests showed that durability of the steel discs significantly increased after the deposition of TiN/ZrN coatings.

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