UDC 539.12.04.621.384.653

SINGLE LAYER AND MULTILAYER VACUUM-ARC COATINGS BASED ON THE NITRIDE TIALSIYN: COMPOSITION, STRUCTURE, PROPERTIES

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Using high-technological vacuum-arc evaporation in the atmosphere of nitrogen with ion bombardment, single- and multilayer coatings based on TiAlSiYN with high mechanical characteristics were obtained: hardness of the coatings reached 49.5 GPA, resistance to wear, with the value of the critical point L_{C5} reaching 184.92 N. The peculiarities of radiation-induced effect at applying bias potential U_b were found: formation of nitride coatings based on fcc metallic lattice with the preferred orientation of crystallites with the texture axis [111], as well as simultaneous growth of hardness. Hardness of both single- and multilayer coatings increases by 40-50 % at the increase of U_b from 50 to 200 V. Formation of silicon-containing layers of TiAlSiYN during the deposition contributes to reaching increased hardness, which, in the case of single-layer coating obtained at U_b = -200 V is 49.5 GPA, which corresponds to super hard state. The mechanisms of structure formation, defining the resulting mechanical characteristics of single- and multi-layer coatings based on TiAlSiYN nitride have been discussed.

INTRODUCTION

Over the past decade, there has been an increased interest to nanostructured materials produced by the vacuum- arc evaporation of metals in the atmosphere of nitrogen [1, 2]. This interest is determined by improving mechanical properties with the decrease of the size of crystallites [3.4]. In vacuum-arc nitride coatings, this effect was most thoroughly studied for single phase TiN material [5]. The obtaining of multielement TiAlN coating on the basis of Titanium Nitride afforded to greatly simplify reaching the nanostructured state and at the same time to increase corrosion strength and wear resistance [6, 7]. Creation of TiAlN coatings showed that by means of introducing new elements and formation of multielement nitrides during this, it is possible to controllably enhance functional properties [8], and at the same time the transition to nanostructured state determines the necessity of tough bonds on the boundaries of crystallites to prevent grain boundary sliding and growth of grains at increased temperatures [9]. For this in some cases artificially created nanostructures, obtained by alternating nitride layers are used [10]. A new way of creation of these type of materials has become obtaining of composites by means of lamination during the dissolution of the thermodynamically unbalanced state of the solid solution at addition of Si as a constituent [11, 12]. This led to the creation of a whole row of highly rigid materials [13–16]. Silicon in these systems is poorly dissolved, and such systems, over-saturated with Si, tend to decay and form composites with high firmness [17, 18].

In this regard, the most promising systems being currently developed include Ti-Al-Si-N [19-21]. The addition of Al component in these coatings led to reassuring results in increased resilience to oxidation, thermal stability and productivity at testing at high speeds of the cutting instrument [22, 23].

Besides, the addition of yttrium atoms to the coating, used in this study, must facilitate the increase the resistance to oxidation due to the formation of YOx phase at the borders of grains [24]. Apart from this, such addition leads to fragmentation of grains and the loss of columness of the structure of condensates, which is important to resist wear by friction in the oxidizing environment.

The aim of this work was studying of the effect of bias potential and thickness of the formed layers (from monolayer, with a thickness of 9 mcm to multilayer with the thickness of layers 12…70 nm) on structure and mechanical properties of vacuum-arc coatings based on nitrides of TiAlSiY alloys.

METHODS OF SAMPLES OBTAINING AND STUDYING

The samples with coatings were obtained by vacuum-arc method on the modernized installation "Bulat-6" [25]. Cathodes of the following composition were made for deposition of multielement coatings: Ti – 58 at. %; Al – 38 at. %; Si – 3 at. %, Y – 1 at. %, and Ti – 62 at. %; Cr – 38 at. %. The cathodes were sintered on installation of spark plasma sintering SPS 25-10.

The addition of operating (nitrogen) atmosphere during the deposition (P_N) was 4.5 10^{-3}Torr. The coating was deposited on the surface of steel 12X18H10T specimens during 2 hours. This allowed obtaining a coating with a total thickness of about 9 µm. Deposition was carried out in both single-layer and multi-layer modes. In the latter case, the coatings were obtained by evaporating 2 cathodes (TiAlSiY and TiCr) with a fixed stopping time of 10, 20, or 40 seconds for each of the 2 cathodes. During the deposition process, a constant negative potential of U_b = -50, -200 or -500 V was applied to the substrate. The arc current (I_a) for both types of cathodes was 100 A, and the focusing current was 0.5 A. The main studies were carried out for the coatings obtained at – U_b = -200 V, the conditions of obtaining of which are shown in Tabl. 1 (in the case of Mode 5, a sublayer of titanium with a thickness of 50 nm was used).

Table 1

Cathodes material and parameters of evaporation and deposition of the coatings with total thickness 9 µm

Cathodes Material	$U_{b,}$ V	Layer deposition time, s	Number of layers
1. TiAlSiY	200	5400	single layer
2. TiAlSiY with Ti sublayer	200	5400	single layer
3. TiAlSiY/TiCr	200	10	533
4. TiAlSiY/TiCr	200	20	270
5. TiAlSiY/TiCr	200	40	135

Phase and structural analysis was carried out on the installation DRON-4 by means of X-ray diffractometry method in Cu-k_α irradiarion. For monochromatization of the recorded radiation, a graphite monochromator, which was installed in a secondary beam (before the detector) was used. The study of the phase composition, structures (texture, substructure) was performed using traditional methods of X-ray diffractometry by analysing the position, intensity, and shape of the profiles of diffraction reflexes. The tables of the International diffraction data center "Powder Diffraction File" were used to decode the diffraction pattern.

The sub-structural characteristics (microdeformation $< \varepsilon >$ and the size of crystallites L) were determined by means of approximation by changing of width of the diffraction reflexes from several reflections degrees [26].

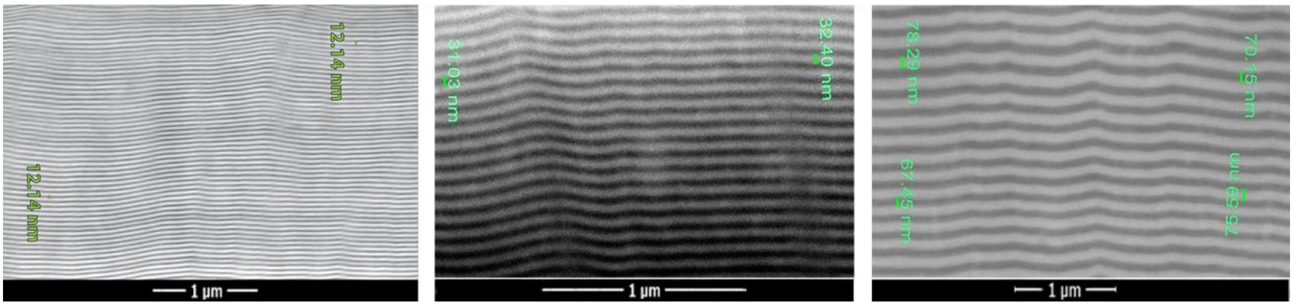
The firmness was measured by means of microindentation method with Vickers Diamond Pyramid as an indenter at loads of 50 g. The study was carried out on the device for microhardness testing 402MVD by Instron Wolpert Wilson Instruments.

The elemental composition of the coatings was studied with by means of electron and ion scanning microscope Quanta 200 3D, the topography of the surface was studied at Nowa Nano SEM 450.

To measure adhesion strength and scratching resistance, scratch-tester Revetest (CSM instruments) was used. The scratches were made on the surface of the coating by diamond spherical indenter "Rockwell S" type, with a radius of curvature of 200 mcm, at a continuously raising load. At the same time, the power of the acoustic emission signal (AE) was detected, friction coefficient and penetration depth of the indenter, as well as normal load were recorded. In order to obtain reliable results, three scratches were made on the surface of each coated sample. The tests were carried out under the following conditions: the load on the indenter was increasing from 0.9 to 70 N, the speed of moving the indenter was 1 mm/min, the length of the scratch was 10 mm, the speed of applying the load was 6.91 N/min, the discrete frequency of the signal was 60 Hz, and the power of the acoustic emission of the signal was 9 dB.

RESULTS AND DISCUSSION

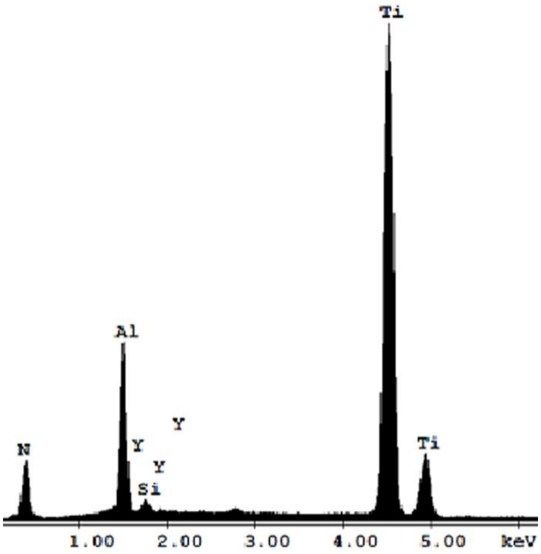
Lateral sections were prepared and electron microscope studies (Fig. 1) were used to determine the thickness of layers obtained in this study at different times of layer deposition. It can be seen that while maintaining good planarity for all deposition conditions, the average thickness of layers during the layer deposition time of 10 seconds (series 3) is around 12 nm (see Fig. 1,a), at the deposition time of one layer of 20 seconds (series 4) is around 32 nm (see Fig 1,b), and at deposition time of the layer of 40 seconds (Series 5) is about 70 nm (see Fig.1,c). Lower relative thickness of growth in the coatings with the thinnest layers can be explained by the large specific volume of the mixed layers and larger relative error in determining the deposition time of the layer. Therefore, it is correct to carry out the most accurate determination of the deposition speed on thick layers. The deposition speed obtained in this manner is about 1.7 nm/s.



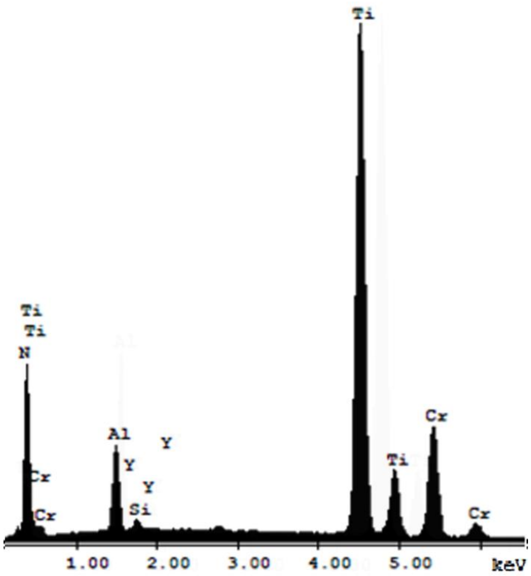
a b c

Fig. 1. Lateral sections of multi-layer coatings obtained at the deposition time of the layer: a – 10 s (series 3), b – 20 s (series 4), and b – 40 s (series 5)

Defining of elemental composition was carried out by energy dispersion method. The characteristic spectra obtained for single- and multi-layer coatings are shown in Fig. 2.



a



b

Fig. 2. Energy dispersion spectra of the coatings: a – monolayer (series 1), b – multilayer (series 4)

The results of element analysis of the coatings obtained during the processing of spectra are shown in Tabl. 2.

Table 2

Data of elemental analysis of the coatings, obtained at different U_b

Series No.	Content of elements, at. %					
	Ti	Al	Si	Y	Cr	N
1	29.75	18.76	1.69	0.43	-	49.45
2	33.21	15.87	1.57	0.51	-	48.84
3	39.62	4.94	0.38	0.06	9.72	45.28
4	40.58	5.26	0.38	-	8.84	44.94
5	40.28	4.52	0.41	-	9.12	45.71

It is seen that the alteration of thickness of layers (series 3–5) has little effect on the element composition. However, the introduction of layers with chromium in the multilayer coating leads to reduction of the relative content of nitrogen atoms in the coating (compare 1, 2 and 3–5 in Tabl. 2).

The obtained coatings consist of nitride phases based on the fcc lattice, as evidenced by the XRD analysis data (Fig. 3). Due to proximity of periods of presumably two components of phases of multilayer coatings, their spectra overlap. Planes in fcc lattice corresponding to the diffraction peaks are marked in Figure 3.

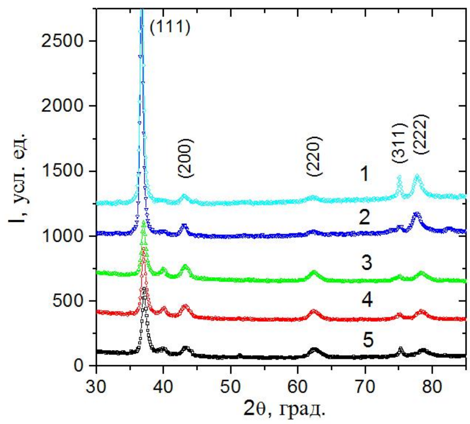


Fig. 3. Areas of X-ray diffraction spectra of the coatings of Series 1 (Spectrum 1), Series 2 (Spectrum 2), Series 3 (Spectrum 3), Series 4 (Spectrum 4), and Series 5 (Spectrum 5)

The obtained specific peculiarity of the obtained spectra should be noted – the shift in the positions of peaks in multi-layer coatings towards large corners in comparison with single-layers. As established in [27], the cause of such shifts could be structural compressive stresses on the interphase boundaries of multi-layer coatings.

The formation of the preferred orientation of crystallites with an axis of [111] perpendicular to the plane of growth (which results in a relative increase in the intensity of the peaks {111} in Figure 3) should be also noted. Its degree of perfection decreases in the multi-layer coatings. Also, the decrease of U_b to -50 V leads to the decrease of the degree of texturedness. In case of the largest U_b = -500 V, a different type of texture with an axis [110] is formed.

The most versatile criterion for mechanical properties is hardness. Figure 4 shows the resulting data for the hardness of different types of coatings. It can be seen that the transition from single layer to multilayer coatings is accompanied by a substantial decrease in hardness.

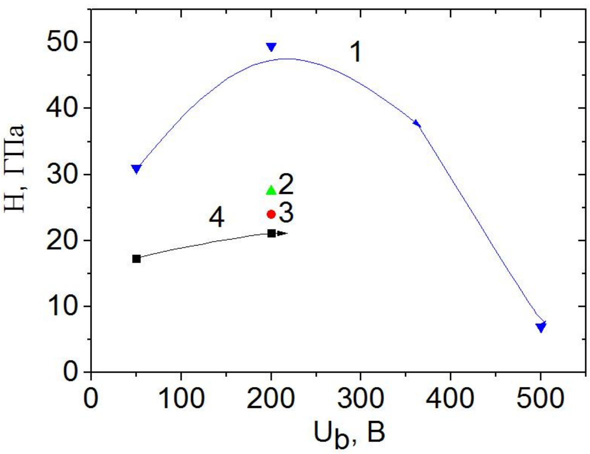


Fig. 4. Dependence of hardness of the coatings on the value of bias potential for single-layer coatings (1); multi-layer coatings with the period of 70 nm (2); with the period of 32 nm (3), and 12 nm (4).

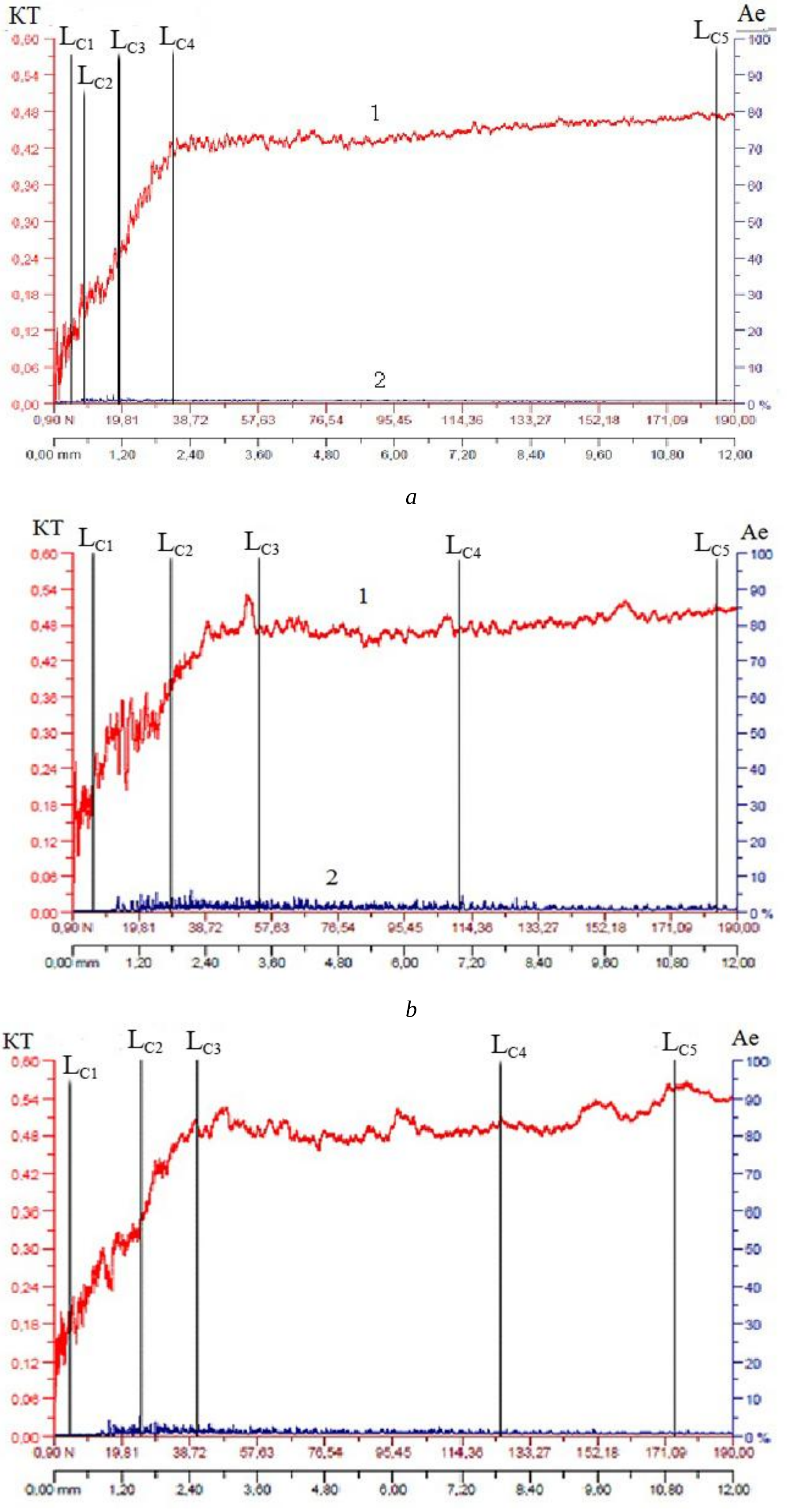
Taking proximity of structural states and phase composition into account, the most accurate reason for such decrease is formation of layers with no Si in multi-layer coatings. Indeed, the standard hardness, reached in nitrides of transition metals based on chrome is 25…30 GPa [28]. At the same time, the obtaining of multielemental nitride coatings with silicon leads to a significantly higher hardness [29]. Decomposition of solid solutions formed during the deposition by sinusoidal type, accompanied by the formation of the composite material of high hardness [12], is considered to be a reason of it, as mentioned at the beginning of the study. Therefore, the hardness in the single-layer state of the Silicon-containing system TiAlSiYN reaches 49.5 GPA.

It should also be noted that the decrease of U_b from -200 V to -50 V leads to a fall of hardness by 40…50% for both single-layer and multilayer compositions. Based on structural data, this decrease can be linked with the transition from sharply textured state with the preferred orientation of crystallites with the axis [111] to the state with almost no texture at U_b = -50 N.

Another important criterion for the coatings is their adhesion strength. A scratch testing method was used in this study to define the adhesion strength parameters. As a result of the tests, friction coefficient for different stages of wear, the amplitude of acoustic emission, and the minimum (critical) loads were determined: L_{C1} corresponds to the beginning of penetration of indenter into the coating, L_{C2} – to the appearance of the first crack, L_{C3} – to the appearance of agglomeration of cracks, L_{C4} – to the delamination of certain areas of coating, L_{C5} – to chip of the coating or its crack resistance coating or its plastic wear to the substrate.

Fig. 5 shows dependence of alteration of friction coefficient (Fc) and the amplitude of acoustic emission (Ae) during scratch testing with an increasing load up to 190 N. It can be seen that acoustic emission for all types of coatings is homogeneous with no extreme peaks, which is corresponds to wear with no brittle fracture. Along with this, in a single-layer coating in a sufficiently narrow load area, the process of cracks accumulation and local laminating (L_{C4}) takes place, and the large area corresponds to wear of the coating down to the substrate (L_{C4} - L_{C5}). In multi-layer coatings, the area is significantly increased to L_{C4}. With the reduction of the period, this change becomes more significant. The friction coefficient at the area L_{C4} - L_{C5} for all systems is about 0.45.

Fig. 6 shows wear grooves in the areas of critical points during load. It is seen that the transition from single-layer coating to multilayer does not lead to qualitative changes in the wear type. Wear in all the areas is uniform, with no explicit chips, which typifies plastic wear. This pattern of wear and tear is observed until the coating is completely worn down to the substrate (see Fig. 6, series L_{C5}).

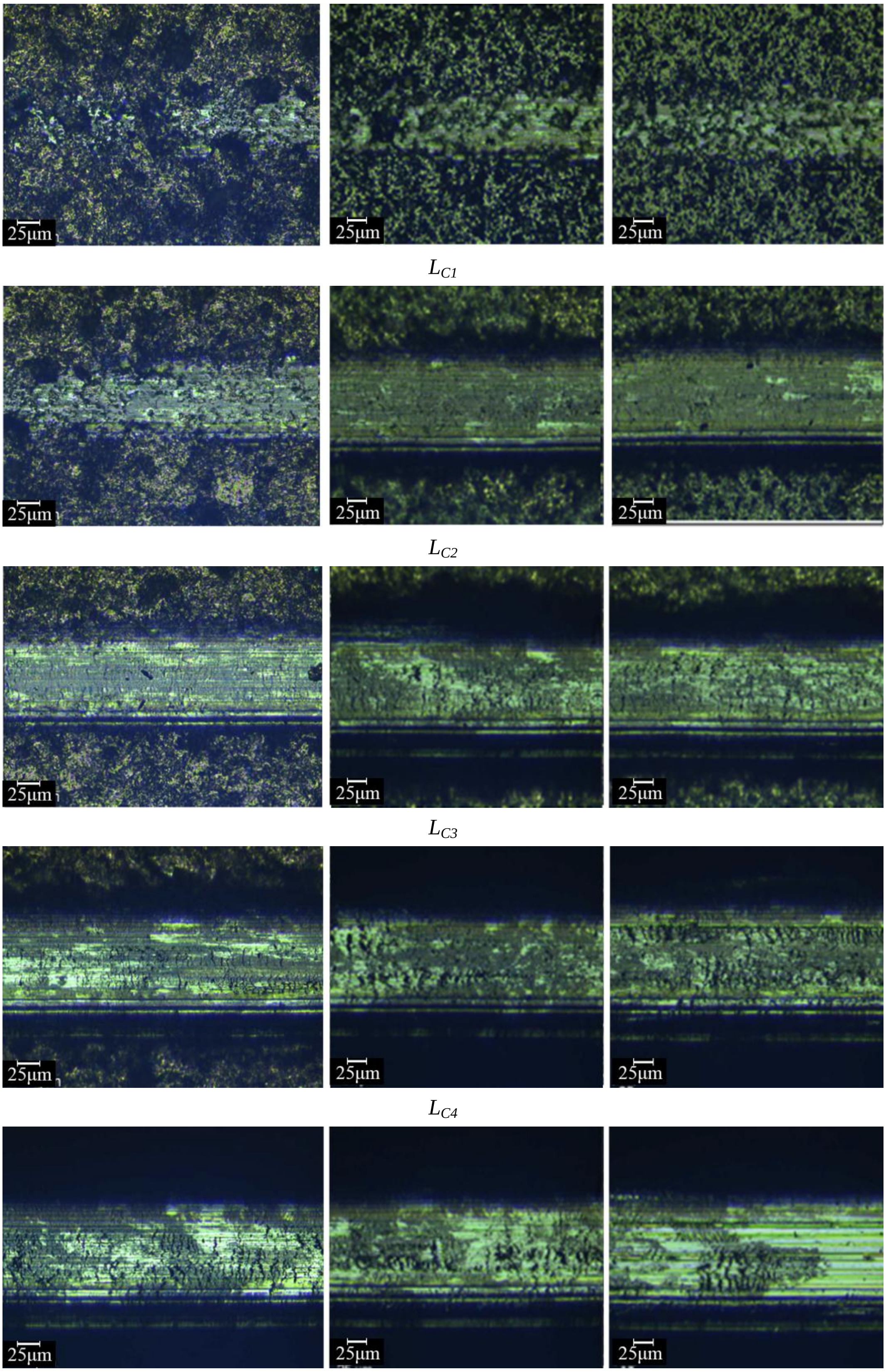


a

b

c

Fig. 5. Changes of the values of the friction coefficient KT (spectrum 1, left scale) and the amplitude of acoustic emission Ae (spectrum 2, right scale) for the coatings, obtained at U_b = -200 V: a – single-layer (series 1); b – multilayer with the biggest period (series 5); c – multi-layer with the smallest period (series 3)



L_{C1}

L_{C2}

L_{C3}

L_{C4}

L_{C5}

a *b* *c*

Fig. 6. Wear grooves in the areas of critical points at a load L_C for the coatings, obtained at U_b = -200 V: a – single-layer (series 1); b –multilayer with the biggest period (series 5); c – multi-layer with the smallest period (series 3)

Tabl. 3 summarizes the results of defining of critical points during loading. It can be seen that single-layer coatings (series 1 and 2), as well as multiperiod coatings with a large period (series 4 and 5), have the highest values of L_{C5}, wich is the evidence of their great efficiency.

Table 3

Critical points L_C at loading the coatings of different series

Series No.	L_C, N				
	1	2	3	4	5
1	5.61	9.32	18.92	34.08	184.92
2	6.85	11.17	24.36	62.19	182.78
3	5.68	25.51	40.79	125.51	173.87
4	5.38	25.36	58.33	100.52	184.87
5	7.05	28.93	54.19	110.81	183.88

At the same time, the initial destruction of multilayer coatings occurs at much higher loads compared with single layers. The reason for such increase in the initial destruction in multi-layer systems is the stop of spreading of cracking development of on interphase boundaries and relaxation of deformation [30].

CONCLUSION

1. Vacuum arc coatings based on TiAlSiYN nitride have high mechanical properties: hardness reaching 49.5 GPA and wear resistance with the value of the critical point L_{C5} reaching 184.92 N.
2. The influence of applied negative bias potential at the deposition affects the formation of the preferred orientation of crystallites: at increase of U_b to -200 V the perfection of the structure of nitrides of the coating with the axis [111] at the metal fcc crystal lattice.
3. The appearance of a texture with an axis [111] is accompanied by a relative increase in hardness of 40…50%, in both single-layer and multi-layer compositions.
4. Formation of silicon-containing layers of TiAlSiYN during the deposition contributes to reaching increased hardness, which, in the case of single-layer coating obtained at U_b = -200 V is 49.5 GPA, which corresponds to super hard state.

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Article received 06.07.2017

ОДНОСЛОЙНЫЕ И МНОГОСЛОЙНЫЕ ВАКУУМНО-ДУГОВЫЕ ПОКРЫТИЯ НА ОСНОВЕ НИТРИДА TIALSIYN: СОСТАВ, СТРУКТУРА, СВОЙСТВА

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Используя высокотехнологичное вакуумно-дуговое испарение в атмосфере азота с ионной бомбардировкой, получили однослойные и многослойные покрытия на основе TiAlSiYN с высокими механическими характеристиками: твердостью, достигающей 49,5 ГПа; стойкостью к износу с величиной критической точки L_{C5}, достигающей 184,92 Н. Выявлены особенности радиационно-стимулированного воздействия при подаче потенциала смещения $U_{см}$: формирование в нитридах покрытий на основе металлической ГЦК-решетки преимущественной ориентации кристаллитов с осью текстуры [111], а также увеличение при этом твердости. При увеличении $U_{см}$ от 50 до 200 В твердость как однослойных, так и многослойных покрытий повышается на 40…50%. Формирование при осаждении кремнийсодержащих слоев TiAlSiYN способствует достижению повышенной твердости, которая в случае однослойного покрытия, сформированного при $U_{см}$ = -200 В, составляет 49,5 ГПа, что соответствует сверхтвердому состоянию. Обсуждены механизмы формирования структуры, определяющие полученные механические характеристики однослойных и многослойных покрытий на основе нитрида TiAlSiYN

ОДНОШАРОВІ І БАГАТОШАРОВІ ВАКУУМНО-ДУГОВІ ПОКРИТТЯ НА ОСНОВІ НІТРИДУ TiAlSiYN: СКЛАД, СТРУКТУРА, ВЛАСТИВОСТІ

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З використанням високотехнологічного вакуумно-дугового випаровування в атмосфері азоту з іонним бомбардуванням були отримані одношарові і багатошарові покриття на основі TiAlSiYN з високими механічними характеристиками: твердістю, що досягає 49,5 ГПа; стійкістю до зношування з величиною критичної точки L_{C5}, що досягає 184,92 Н. Виявлено особливості радіаційно-стимульованого впливу при подачі потенціалу зміщення $U_{зм}$: формування в нітридах покриттів на основі металевих ГЦК-граток переважної орієнтації кристалітів з віссю текстури [111], а також збільшення при цьому твердості. При збільшенні $U_{зм}$ від 50 до 200 В твердість як одношарових, так і багатошарових покриттів підвищується на 40…50%. Формування при осадженні шарів TiAlSiYN, що містять кремній, сприяє досягненню підвищеної твердості, яка в одношарового покриття, отриманого при $U_{зм}$ = -200 В, становить 49,5 ГПа, що відповідає надтвердому стану. Обговорено механізми формування структури, що визначає отримані механічні характеристики одношарових і багатошарових покриттів на основі нітриду TiAlSiYN.