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**FUNCTIONAL COATINGS  
AND SURFACE TREATMENT**

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## Structure and Mechanical Properties of TiAlSiY Vacuum-Arc Coatings Deposited in Nitrogen Atmosphere

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**Abstract**—The effect of a negative bias potential applied to the substrate on elemental composition, structure, and mechanical properties of vacuum-arc TiAlSiY nitride coatings is investigated by different methods. It is ascertained that applying a high (up to  $-500$  V) bias voltage leads to a selective sputtering of target as well as significant microdeformation of the coating, small-sized growth of crystallites, and their preferred orientation along the [110] direction. In this case, the coating deposited has a low hardness  $H = 6.95$  GPa and propensity to intense wear under scratch and tribological tests. Crystallites with stoichiometric composition  $\sim 140$  nm in size and [111] preferred orientation perpendicular to the surface of growth are formed at the bias potential about  $-200$  V, and superhard ( $H = 49.5$  GPa) and wear-resistant coatings are grown under such conditions. The mechanisms of formation of the structure of multielement coatings are discussed. It is shown that formation of an amorphous phase and nanocrystal [110] texture takes place at a high bias potential owing to the process of radiation-stimulated selective spraying of the target. Formation of microstrained crystallites 10 nm in size caused by the weakening of interatomic bonds is observed in the coating under such deposition conditions, and it leads to the decrease in coating hardness and fast destruction during tribological testing. Substantial bias voltage dependence of deposited coating properties is established in our investigations.

**Keywords:** vacuum-arc deposition, wear, friction coefficient, surface, structural engineering, solid solution

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### INTRODUCTION

It is known that the operational characteristics of functional materials, such as fatigue strength and wear

and corrosion resistance, depend on the structural features of the surface layer [1, 2]. In order to increase the operational life of articles, an increase in the qual-

ity of the bulk properties of the material is usually not required; it is sufficient to modify the surface layers [3, 4] and this can be achieved by depositing coatings based on compounds of materials with tailored properties [5, 6].

Growth of requirements on the reliability of equipment under intense thermobaric loads determines further improvement of the materials of coatings and procedure for their deposition [7]. One of the ways to solve this problem is the design of multicomponent and multilayer coatings, which are used, for example, to increase the performance of the details of cutting tools which operate at high cutting speed, to increase the reliability of friction units, and to protect the details against corrosion [8].

High thermal stability of physicomaterial properties and high temperature stability to oxidation, as well as low adhesion activity, are important characteristics of the coating, which significantly depend on its phase composition and thermal stability of individual phases or layers which form the coating [9, 10].

Recently, particular attention has been devoted to multielement nitride systems involving silicon as one of the components [11, 12]. Silicon dissolves poorly in nitride systems of transition metals and these silicon-oversaturated systems tend to destruct to give composites with high hardness [13, 14]. The system of (TiAlSi)N is one of the most promising. The use of Al in these coatings provides their enhanced resistance to oxidation, thermal stability, and high operational characteristics of a cutting tool [15, 16].

Addition of yttrium to the coating should provide an increase in its resistance to oxidation owing to the formation of the  $YO_x$  phase at grain boundaries [17]. In addition, this additive leads to dispersion of grains and inhibition of the formation of a columnar structure of condensates, which is essential from the viewpoint of the increase in the friction wear resistance in an oxidative medium [18].

Most works on the design of nitride coatings based on the TiAlSi system employ ionic-plasma magnetron sputtering [19] using a mixture of Ar/N<sub>2</sub> gases as the sputtering atmosphere. In this case, at the content of Si less than 8 at %, the coating with the structure of a single-phase solid solution with the preferential orientation of crystallites along the [111] texture axis, which possesses high hardness achieving the value of 42 GPa at the maximum Si content of 8 at %, is formed. However, the highest compressing stresses (up to 11 GPa) develop in the coatings with the Si content of around 4 at % [19] and the hardness values remain almost unchanged at high-temperature vacuum annealing up to the temperature of 1000°C.

The reasons for the achievement of high physicomaterial properties in the surface layers and the pos-

sibility of the controlled change of these properties are still investigated insufficiently. Although, the methods for physical vapor-phase deposition (PVD) at low temperatures, which also include magnetron sputtering, are very popular and are widely used to prepare coatings of this type, low temperature imposes significant kinetic restrictions hindering the formation of oversaturated solid solutions in the metastable state [6, 9].

In this work, in order to prepare superhard wear-resistant coatings, vacuum-arc deposition was employed, which provides an increase in the mean energy of deposited particles. In this case, the degree of ionization of the plasma achieves 90%, which makes the plasma fluxes controllable and gives high-density coatings of large thickness (up to 10 μm) within the process time of deposition of 60 min [7, 20]. The aim of this work was to determine the effect of the bias potential applied to the substrate on the composition, structure, and mechanical properties of nitride vacuum-arc coatings prepared by the deposition from an alloy metal cathode of the TiAlSiY system in a nitrogen atmosphere.

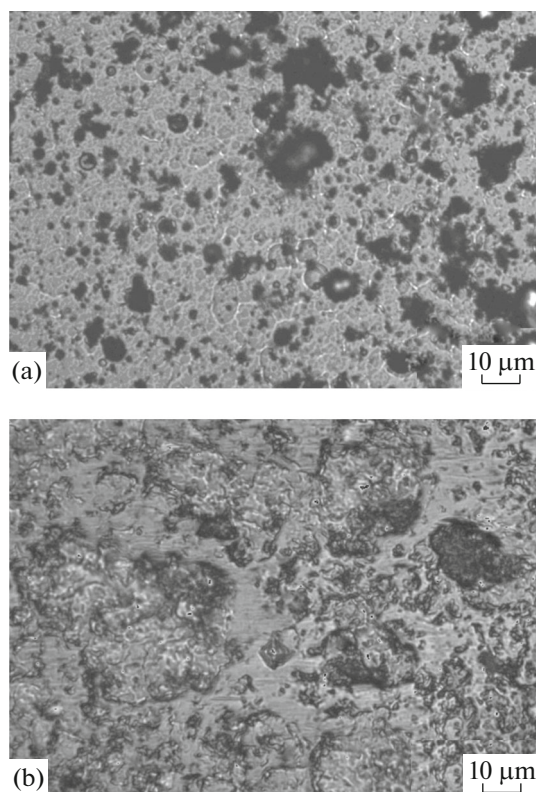
## PROCEDURE OF EXPERIMENT

The specimens were prepared using the vacuum-arc method on a Bulat-6 modernized setup [20]. In order to deposit multilayer coatings, a cathode with the composition 58 Ti–38 Al–3 Si–1 Y (at %) composition was used. The cathode was sintered on a SPS 25-10 setup for spark plasma sintering.

The pressure of the working atmosphere (nitrogen) during deposition was  $P_N = 5 \times 10^{-3}$  Torr. Deposition was carried out from one source for 2 h, which provided the coatings with the thickness of ~9 μm. During deposition, a constant negative bias potential  $U_b = -200$  V (batch 1) or  $U_b = -500$  V (batch 2) was applied to the coating.

The structural-phase analysis of the specimens was carried out using X-ray diffractometry in Cu  $K_\alpha$  radiation on a DRON-4 instrument. To monochromatize the recorded radiation, a graphite monochromator was used, which was mounted on a secondary beam in front of the detector. The phase composition and structure (texture, substructure) of the coatings were studied using conventional methods of X-ray analysis. To resolve the diffractograms, the tables of the International Center of Diffraction Data Powder Diffraction File were used. Separation of the profiles of diffraction peaks into components was performed using the NewProfile program package [21].

Substructural characteristics (microdeformation  $\epsilon$  and crystallite size  $L$ ) were determined by approxima-



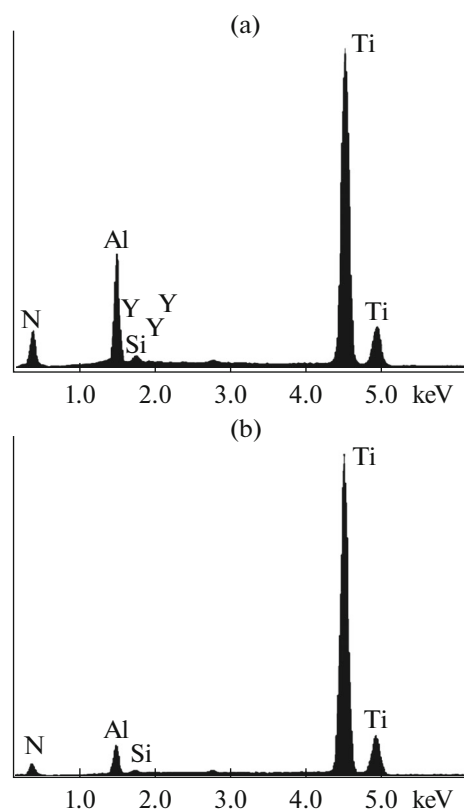
**Fig. 1.** Surface morphology of the polished (TiAlSiY)N coatings deposited at the substrate bias potential  $U_b =$  (a)  $-200$  V and (b)  $-500$  V.

tion according to the change of the width of diffraction reflexes from several orders of reflections.

Elemental composition of the coatings was studied using a Quanta 200 3D focused ion beam/scanning electron microscope; the surface topography was studied on a Nova NanoSEM 450 microscope.

Adhesion strength and resistance of the coatings to scratching were determined using a Revetest scratch tester (CSM Instruments) and microhardness of the coatings was measured on an AFFRI DM-8 automatic Vickers hardness meter at the load on the indenter of 50 g.

Tribotechnical tests of the coatings were carried out according to the standard “ball–disk” diagram on a high-temperature tribometer (CSM Instruments) using factographic analysis of the wear groove on the coating and the wear spot on a countersolid (ball made from corundum  $\text{Al}_2\text{O}_3$  with the diameter of 6 mm). Wear resistance tests were carried out in the air at the load of 5 N and the linear rate of 15 cm/s; the bending radius of wear was 5 mm and the sliding distance was 500 m. Groove depth measurements were carried out in four diametrically and orthogonally opposite ranges of the specimens using a Surtronic 25 automatic precision contact profilometer; in this case, the mean area



**Fig. 2.** Energy dispersion spectra of the (TiAlSiY)N coatings: (a) batch 1,  $U_b = -200$  V; (b) batch 2,  $U_b = -500$  V.

of the cross section and wear groove depths were determined.

## RESULTS AND DISCUSSION

The surface morphology of the coatings of the (TiAlSiY)N coatings prepared at various bias potentials  $U_b$  (batches 1 and 2) differs according to the homogeneity of surface formations (Fig. 1) After polishing, in order to remove the drop phase, it is clear that a relatively homogeneous structure of the polished section of the coating prepared at relatively small  $U_b = -200$  V (Fig. 1a) transfers to a heterogeneous structure with an increase in the bias potential to  $U_b = -500$  V (Fig. 1b), where the regions with clear fusion are observed, which can be related to the high local temperature in the range of multistage defect formation at higher energies of the deposited particles [22].

Radiation-stimulated processes during the bombardment of the deposited coating at higher bias potentials  $U_b = -500$  V on the substrate affect its composition owing to the selective sputtering of light and weakly bonded particles. In Fig. 2, intrinsic energy-dispersion spectra are given, while in Table 1, the elemental composition of the coatings calculated accord-

**Table 1.** Results of elemental analysis for the coatings deposited at different values of the substrate bias potential  $U_b$ 

Batch	Content, at %				
	Ti	Al	Si	Y	N
1	29.75	18.76	1.69	0.43	49.45
2	77.59	7.78	0.79	—	13.84

**Table 2.** Critical points  $L_C$  under the loading of coatings deposited at different values of substrate bias potential  $U_b$ 

Batch no	$L_C$ , H				
	1	2	3	4	5
$a$	5.61	9.32	18.92	34.08	184.92
$b$	6.41	18.99	22.46	35.20	70.78

ing to these spectra is given. It is clear that, in the coatings obtained at  $U_b = -200$  V (batch 1), the entire elemental composition of the cathode is revealed, while the nitrogen content is close to 50%, which indicates the stoichiometry of mononitride.

In the second series of experiments, a significant depletion of light Al and Si elements is observed in the coatings prepared at  $U_b = -500$  V; yttrium is not detected, while the nitrogen content decreases significantly. These changes can be related to the ballistic sputtering of elements, at which lighter and weakly bound atoms are predominantly removed.

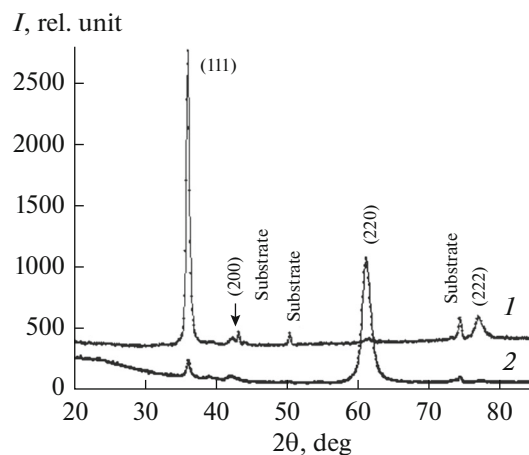
In diffraction spectra, the mentioned changes of elemental composition lead to the shift of peaks and

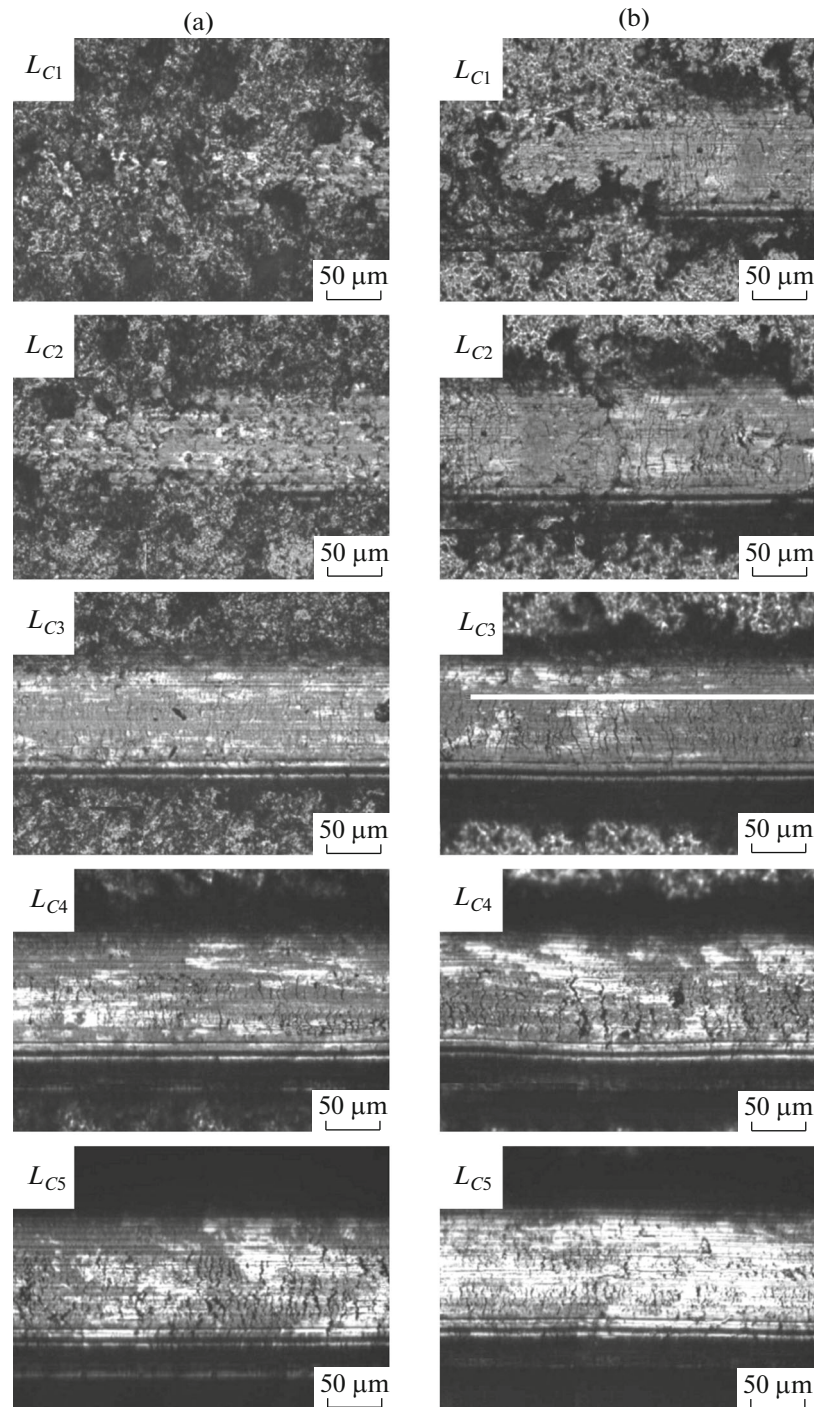
the change of the predominant orientation of crystallites (texture) (Fig. 3). In this case, in the specimens of both batches 1 and 2, a single-phase state is detected with an fcc crystal lattice. The lattice period in the case of the specimens of batch 1 ( $U_b = -200$  V) is  $a = 0.4271$  nm, while in the case of the specimens of batch 2 ( $U_b = -500$  V),  $a = 0.4249$  nm.

The difference in bias potentials and, consequently, the energy of deposited particles affects the formation of the predominant orientation of crystallites. At  $U_b = -200$  V, the texture with the [111] axis is formed, which is perpendicular to the growth plane (in spectrum 1, Fig. 3, this manifests itself in the relative increase in the reflex intensity of {111}). The formation of this texture corresponds to the arrangement of the most densely packed layers from metal atoms and nitrogen atoms in parallel to the growth plane [20].

At the higher bias potential  $U_b = -500$  V, the texture with the [110] axis is formed, which is determined by the lowest free energy at radiation-stimulated multistage formation [23]. In addition, at the higher bias potential, an amorphous-like phase is formed in the coating, which manifests itself in diffraction spectrum 2 (Fig. 3) as a “halo-shaped” curve at small angles with the center of  $2\theta \approx 22^\circ$ .

At a substructural level, the effect of high bias potential manifests itself in an increase in the microdeformation to  $\langle \epsilon \rangle \approx 1.7\%$  and a decrease in the size of crystallites to  $L \approx 11.7$  nm. The reason for the increase in microdeformation with an increase in the bias potential to  $U_b = -500$  V can be related not only to the more intense radiation defect formation but also to a significant depletion of the coatings of batch 2 in nitrogen, because the formation of vacancies in the tetrahedral nodes occupied with nitrogen atoms provides a more significant microdeformation of the crystal lattice. For comparison, at  $U_b = -200$  V, in the

**Fig. 3.** Parts of the X-ray diffraction spectra of (TiAlSiY)N coatings of (1) batch 1 and (2) batch 2.



**Fig. 4.** Wear tracks in the areas of critical points under loading  $L_C$  for (TiAlSiY)N coatings deposited at the substrate bias potential (a)  $U_b = -200$  V and (b)  $U_b = -500$  V.

coatings of batch 1, the values of  $\langle \epsilon \rangle \approx 0.64\%$  and  $L \approx 142$  nm were recorded.

The differences in the structural-phase state are also determined by significant differences in mechanical properties of the coatings prepared at various bias potentials  $U_b$ .

In the first series of experiments ( $U_b = -200$  V), the coatings are characterized by the superhard state ( $H_{0.05} = 49.5$  GPa) possessing high adhesion strength and resistance to crack formation (Fig. 4a, Table 2), as well as relatively low friction coefficient  $\mu = 0.42$  (Fig. 5a). The wear of the coating at tribological tests

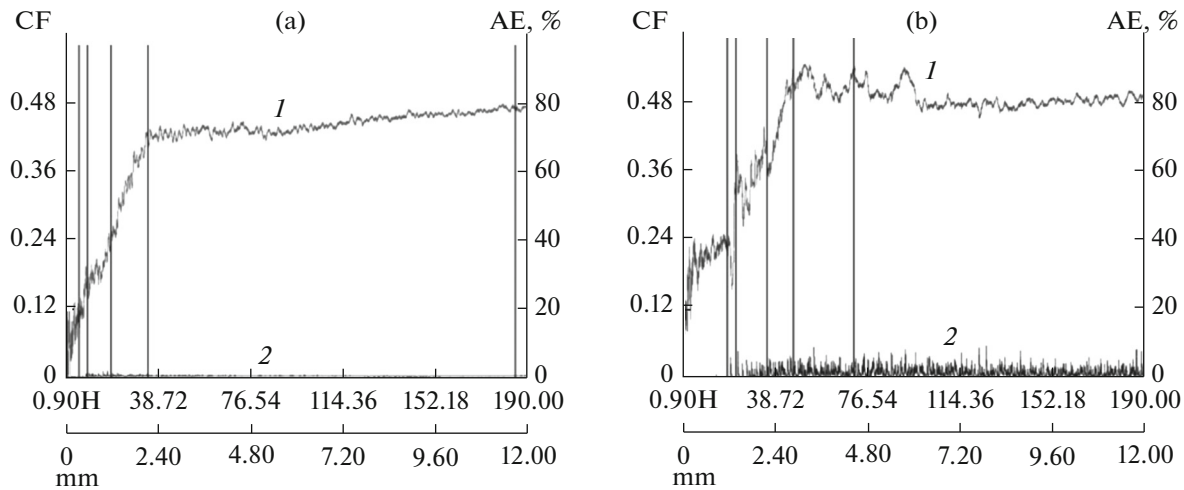


Fig. 5. Values of the coefficient of friction CF (1, left scale) and amplitude of acoustic emission AE (2, right scale) under scratching test of (TiAlSiY)N coatings deposited at the substrate bias potential (a)  $U_b = -200$  V and (b)  $U_b = -500$  V.

(Fig. 6) conducted at the load of  $F = 5$  N (friction path  $S = 500$  m and radius of curvature of wear  $r = 5$  mm) is also relatively low. During the tests, the (TiAlSiY)N coating preserved its integrity and was not torn down to the substrate, while the wear values of both the coating and the countersolid were low and corresponded to  $1.06 \times 10^{-5}$  and  $9.03 \times 10^{-7}$  mm<sup>3</sup>/N m, respectively.

In the (TiAlSiY)N coatings prepared at high bias potential  $U_b = -500$  V (batch 2), there is a decrease in hardness to 6.95 GPa owing to the nonstoichiometry on nitrogen and radiation-stimulated structural (formation of an amorphous-like phase and appearance of a labyrinth pattern on the surface, which is intrinsic to an amorphous-like state) and substructural changes (large values of  $\langle \epsilon \rangle$  and low values of  $L$ ), which leads to the enhancement of wear at large loads ( $L_{C4}$  and  $L_{C5}$  in Fig. 4b) and low critical load of destruction  $L_{C5}$  (Table 2). The friction coefficient in this case increases to 0.5 and becomes inhomogeneous. The amplitude of acoustic emission corresponding to dislocation releases of deformation also increases (Fig. 5b), while at tribological tests even at low load of 2 N, the coating is torn down to the substrate.

Thus, multielement nitride coatings of the (TiAlSiY)N system prepared using vacuum-arc deposition are very sensitive to the value of the negative bias potential  $U_b$  applied to the substrate. At high bias voltages  $U_b = -500$  V, the amorphous-like phase and the texture of nanocrystallites with the [110] axis are formed owing to the intense radiation-stimulated selective sputtering of the surface of the coating. In this case, crystallites of small sizes are formed and there is a strong microdeformation of the crystal lattice because of the weakening of atomic bonds. All this leads to a decrease in the hardness of the coating and

worsening of its tribological characteristics. High wear of the coating results in its destruction even at relatively low critical loads.

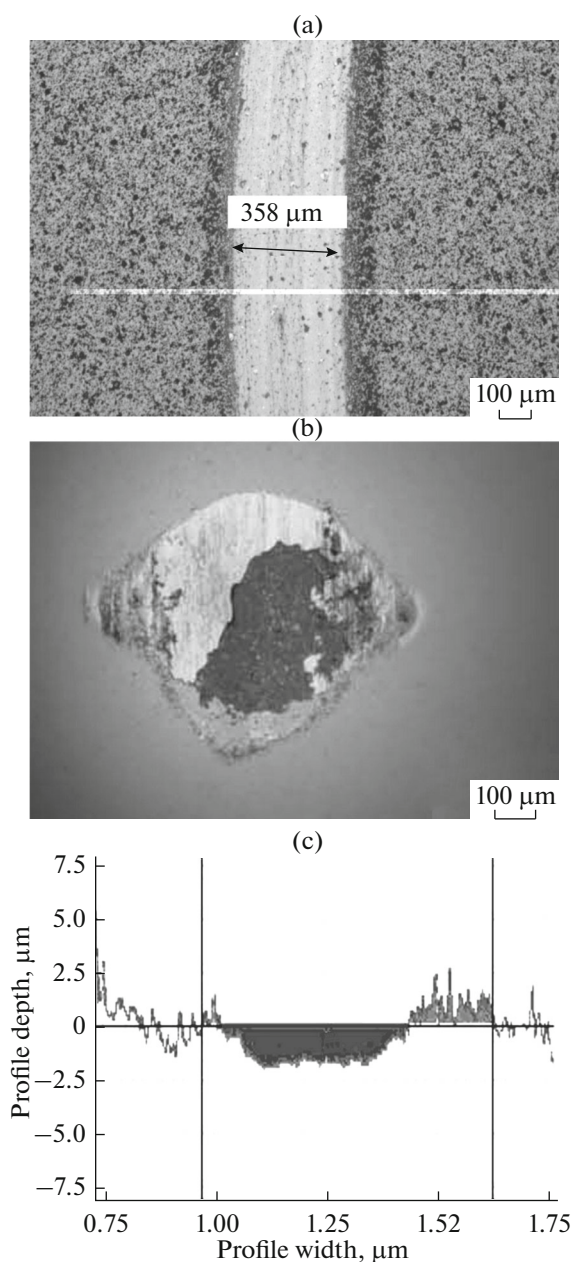
A decrease in the bias potential to  $U_b = -200$  V is optimal not only for the formation of TiN mononitride [24] but also from the viewpoint of the increase in mechanical properties and wear resistance of multielement nitride coatings based on titanium. This is caused to a significant extent by their elemental composition, which is close to stoichiometric, as well as by the formation of the structure with the axis of texture of crystallites [111], low microdeformation of the crystal lattice, and medium size of crystallites of  $\sim 140$  nm.

## CONCLUSIONS

(1) Using vacuum-arc deposition through sputtering of the integral-cast multicomponent TiAlSiY cathode, nitride coatings have been prepared, whose features depend significantly on the value of the negative bias potential applied to the substrate during the deposition of the coating.

(2) It has been shown that the value of the bias potential affects the preferential orientation of the crystallites. At the bias potential of  $U_b = -200$  V, the texture with the [111] axis is formed, which is perpendicular to the growth plane, while at  $U_b = -500$  V, the texture with the [110] axis is formed, which is determined by the lowest free energy during radiation-stimulated cascade formation.

(3) Nitride (TiAlSiY)N coatings prepared at  $U_b = -200$  V possess high hardness  $H = 49.5$  GPa, while the hardness of the coatings prepared at  $U_b = -500$  V decreases to  $H = 6.95$  GPa. The effect of the bias potential on the hardness is rationalized by the more



**Fig. 6.** Wear track on the (a) surface of coating and (b) Al<sub>2</sub>O<sub>3</sub> ball counterbody and (c) profile of the wear groove of the coating after testing of (TiAlSiY)N coating (batch 1).

intense formation of radiation defects at high voltage applied to the substrate, as well as a significant depletion of the deposited coating in nitrogen atoms.

(4) At the bias potential  $U_b = -200$  V, wear-resistant nitride coatings are formed, whose wear is less than  $1.06 \times 10^{-5}$  mm<sup>3</sup>/N m at tribological tests at the load of 5 N (wear of the countersolid in this case is  $9.03 \times 10^{-7}$  mm<sup>3</sup>/N m). In the coatings prepared at  $U_b = -500$  V, tearing down to the substrate is observed under analogous tests already at the load of 2 N.

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