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Structure and Physics Mechanical Properties of Multiperiod Vacuum-arc Coatings on the Basis of Two-layer System TiN_x/ZrN_x

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By methods of structural analysis (a precise X-ray diffraction method and raster electron microscopy) in conjunction with tests on physical and mechanical characteristics (hardness, elastic modulus, friction force and friction coefficient) comprehensive studies have been conducted. Such complex researches are the basis for optimization of properties of multiperiod systems TiN_x/ZrN_x by changing their structural states (structural engineering). The main parameters of changes were: number of layers (n) from 134 to 534 (at total coating thickness of about 10 microns) and magnitude of negative bias potential U_b. Formation of biphasic (TiN_x and ZrN_x) condition was revealed. On substructural level the most sensitive to U_b is micro-strained state. The growth of micro-strain with an increase numbers of ZrN_x layers (at the greatest $U_b = -200$ V) testifies about the determining contribution of irradiation of heavy Zr ions in defect formation at formation of coating. Is established that under optimal technological parameters of receiving of multiperiod TiN_x/ZrN_x coatings their hardness is in the range 40-50 GPa that corresponds to super hard condition. Dependence of penetration depth of indenter is revealed during testing in a pair of "diamond - multiperiod TiN_x/ZrN_x coating" and coefficient of friction on the ratio H/E, which characterizes the elasticity of material.

Keywords: Multiperiod coating, Number of layers, TiN_x/ZrN_x, Substructure, Micro-strain, Crystallite size, Hardness, Depth of penetration, Friction coefficient.

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1. INTRODUCTION

Is known that functional characteristics of materials, such as fatigue strength [1, 2], wear- [3, 4] and corrosion resistance [5] etc. depends on features of structure of surface layer [6, 7].

For increase resource of operation products is usually not required increase of bulk properties their materials, is sufficient surface modification of material [8, 9]. To the greatest degree this can be achieved through application of coating on the basis of composite materials with special properties [10-12].

Such coatings are widely applied in modern technique. Enhancing of requirements to reliability of techniques in conditions intensive thermobaric load causes necessity of further improvement of coatings [11, 12]. One way of solving this task is creation of multicomponent [13] and multilayer [9] coatings that used to improve operability details of cutting tools (operating at high speeds of cutting), improving the reliability of friction units, protection of details against corrosion [14, 15].

High thermal stability of physical and mechanical properties [16] and high sustainability to oxidizing of [17], low adhesion activity [18] are necessary properties of mono- and multilayer coatings [19]. These properties are strongly dependent against phase composition, thermal stability of separate phases (for layers, out of which comprised the coatings) [20].

However, questions that associated with processes and mechanisms of achieving high physical and mechanical properties in coatings and possibility to manage these properties yet require answers and explanations.

In recent time all larger attention is given to coatings from zirconium nitride. Availability of own base of zirconium in Ukraine, its high physical and mechanical properties and great use in atomic energy open wide prospects for its industrial application in the form of vacuum-arc coatings. Particularly high properties demonstrate multiperiod compositional coatings on the basis of zirconium nitride: ZrN/MoN [21], ZrN/CrN [22, 23] ZrN/TiN [24].

2. SAMPLES AND METHODS OF RESEARCHES

Multilayered two-phase nanostructured coatings TiN_x/ZrN_x were precipitated in vacuum-arc installing "Bulat-6" [25]. By way of materials of cathodes are used: titanium BT 1-0; low-alloy zirconium; active gas – nitrogen (99,95 %). Coatings were deposited on surface of the samples (20 × 20 × 2 mm) from steel 12X18H10T that prepared by standard methods of grinding and polishing. Procedure of deposition of multilayer coatings is included following operations. Vacuum chamber was evacuated to a pressure of 10^{-5} Torr. Then to swivel apparatus with substrate holder were fed negative potential of 1 kV, were included evaporator and were produced purification of surface of first of the two substrates by bombardment of ions of chromium during 3 ... 5 min. Thereafter substrate holder was rotated 180° and was carried out same purifi-

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cation of second substrate. Further concurrently were included are both evaporators, was fed nitrogen into the chamber and were precipitated first layer from one side ZrN, and from the opposite – TiN.

Process of deposition was performed at the following technological conditions. After deposition of first layer are both evaporators were turned off, were turning substrate holder for 180° and again concurrently have included both evaporators. Arc current during the deposition was 100 A, nitrogen pressure (P_N) in the chamber was varied in the interval $10^{-5}…5 \times 10^{-3}$ Torr, distance from the evaporator to the substrate – 250 mm, substrate temperature (T_s) was in the interval 250 ... 350° C. Were obtained coatings of thickness about 10 microns. At the time of deposition on a substrate was fed constant negative potential $U_b = -70 ... -200$ V.

Multilayer nanostructured coatings TiN_x/ZrN_x with simultaneous ion implantation in the deposition process were precipitated at filing on substrate holder of negative potential, both in a constant and in the pulsed modes (with a pulses duration 10 microseconds, frequency of following 7 kHz and amplitude (U_{ip}) up to 2000 V) [25]. The main advantage of this method of plasma ion implantation and deposition (PBII&D-method) during synthesis of two-phase nanostructures TiN_x/ZrN_x is to substantially reduce of temperature substrate (below 200° C) that should block noticeable diffusion stirring of system components [26, 27].

Phase composition, structure and substructural characteristics have been studied by method X-ray diffractometry (DRONE-4) with use Cu-K_α-radiation. For monochromatisation of registered radiation was used graphite monochromator, which was installed in a secondary beam (ahead of the detector). Study of phase composition, structure (texture, substructure) were produced by means traditional methods of ray diffractometry through the analysis of position, intensities and forms of profiles of the diffraction reflexes. For decryption of diffractograms were used tables of International Centre for Diffraction Data Powder Diffraction File. Substructure characteristics were determined by method approximation [10, 20].

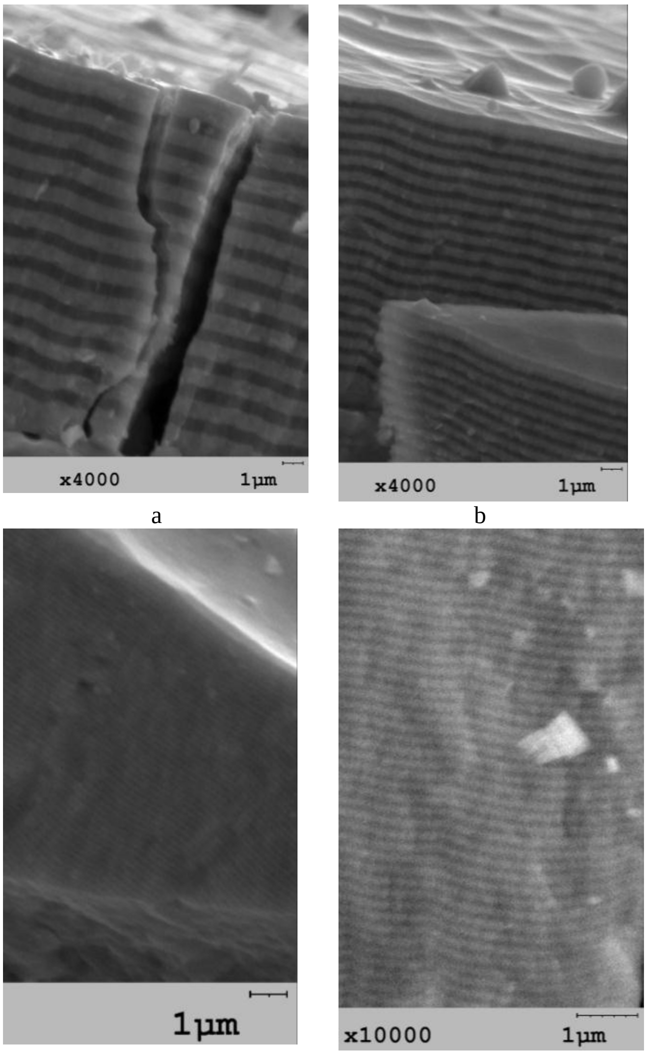
Microindentation was performed on installing "Micron-gamma" at a load till $F = 0{,}5$ H diamond pyramid Berkovich with an angle of sharpening 65°, with automatically execution loading and unloaded throughout 30 seconds [28].

Research of characteristics wear resistance was performed on the device "Micron Friction" by method of rotation of diamond indenter with radius of curvature of about 500 microns on a circle of covering. The load under friction was chosen such that the thickness of immersion indentor has been less than coating thickness. The load was varied in the range of 150-550 g and the number of friction circles has reached 1000.

In the process of friction were taken photos of the friction surface, the curve of the scanning depth of the groove and 3D profile of the groove itself. In automatic mode were fixed the load level (P) and the friction force between diamond surface and the coating (F). The friction coefficient (f) was determined as a result of attitude of friction force to the load.

3. RESULTS AND DISCUSSION

As have shown researches of microstructure of kinks – multilayer coating even when applying a large bias potential of – 200 V that leads to high energy of charged film-forming particles, retains good planarity of deposited layers without express large droplets (between the layers and within the layers) (Fig. 1).



a b

c d

Fig. 1 – The microstructure of the fracture of multilayer coatings TiN_x/ZrN_x, that obtained by $P_N = 0{,}4$ Pa and an action $U_b = -200$ B with the number of layers: a – 134 (bilayer thickness $\Lambda \approx 800$ nanometers), b – 268 ($\Lambda \approx 400$ nanometers), c, d – 534 layers ($\Lambda \approx 200$ nanometers) at different magnification. Total coating thickness about 10 microns

More light layers – ZrN_x, which is receive approximately 1.5 times thicker than TiN_x. That is deposition rate TiN_x is lower than of nitride zirconium.

Method of ray diffractometry been used for study of phase composition, structure and substructural states. According to results of decipherment data of X-ray diffraction spectrums (Figure 2) for all used periods and the thicknesses are revealing 2 systems of diffraction spectrums: one is inherent to TiN_x phase (JCPDS 87 – 0629 card), the second – ZrN_x phase (JCPDS 35 – 0753 card).

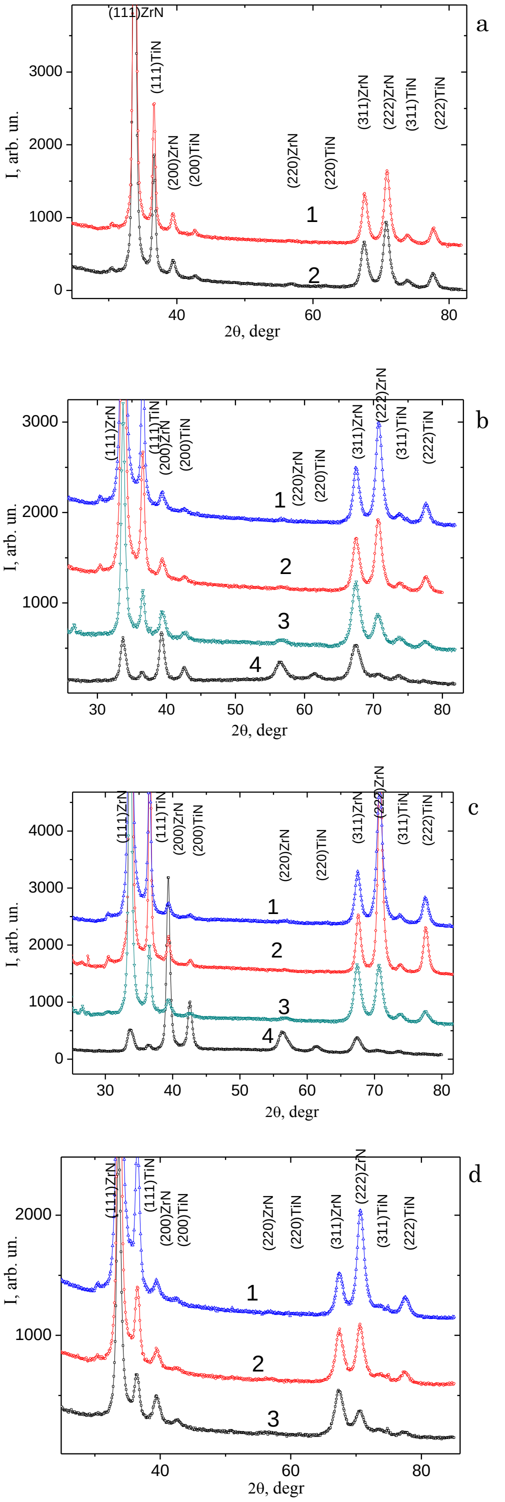


Fig. 2 – Plots of diffraction spectra of coatings with different numbers of layers 134 (a), 268 (b), 359 (c) and 534 (d) and obtained at transmitting a negative potential to the substrate U_b: 1 – 200 V, 2 – 150 V, 3 – 70 V, 4 – 70 V and $U_{ip} = -2000$ V

As is evident from diffraction spectrums of coatings that obtained with the least number of layers $n = 134$ at $\Lambda \approx 800$ nm (Figure 2, a), the use U_b in the interval – 140 ... – 200 V does not lead to noticeable changes in phase-structural condition of coatings. Same is possible to state also for a larger $n = 268$ (Figure 2b, spectrums 1 and 2). Comparative researches conducted for this series of coverages with using U_{ip} are showed (spectrums 3 and 4, Figure 2, b) that using of high voltage potential leads to a decrease degree texturing with axis of texture [111] and the emergence of yet two other axes of texture [311] and [110]. Let us note that the formation of axis of texture [110] determined by the action of radiation factor (cascade-forming) at deposition [25, 29, 30].

More significant is changed texture at applying U_{ip} in coatings with a larger number of layers $n = 359$ ($\Lambda \approx 200$ nm). A strong texture [111] inherent to deposition with $U_b = -70 ... -200$ V (Figure 2, c, spectrums 1, 2, 3) is changed in ZrN_x layers on [110] (Figure 2, c, spectrum 4). In TiN_x layers the texture [111] is retained.

At the largest number of layers 534 (Figure 2, d) is happening strengthening of texture [111] in the layers TiN_x and ZrN_x.

On substructural level the largest effects in changing of dislocation structure – at $U_b = -200$ V (Figure 3). Change in mean size of crystallites (the ordered dislocation) is happening stronger in ZrN_x layers. In comparison with TiN_x layers in ZrN_x layers is more the middle size of crystallites by absolute meaning. This may be connected with the fact that system Zr-N has a much more stronger bonds between atoms (high heat of the formation) than TiN_x.

At a large number of layers and their lesser thickness the middle sizes of crystallites in both layers are becoming approximately identical. This quantity *L* is determined by thickness of the layer.

In layers of coatings obtained at a lower $U_b = -140$ V (Figure 3, c) tendencies that above described are stored.

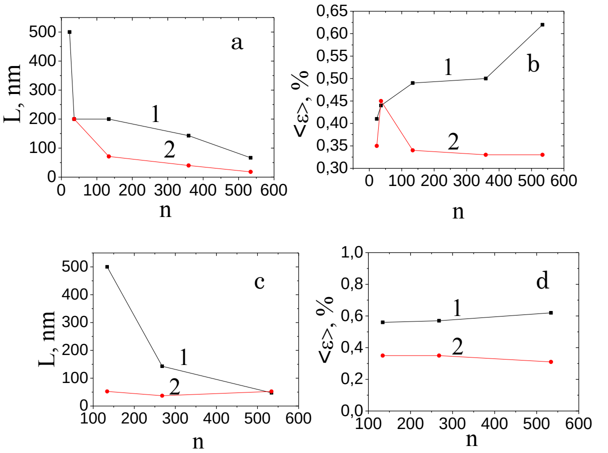


Fig. 3 – Substructure characteristics (*L* – size of crystallites, <*ε*> – micro-strain) ZrN_x (1) and TiN (2) layers of coatings by thickness about 10 microns, depending upon number of layers n: a, b – $U_b = -200$ V, c, d – $U_b = -140$ V

The greatest impact of U_b on the micro-strained state (Figure 3, b, d). For comparatively lower $U_b = -140$ V the absolute values <*ε*> practically independent of number of layers (and respectively of their thickness). At larger $U_b = -200$ V and small thicknesses of ZrN_x layers (large *n*) – they strongly micro-strained (high density of disordered dislocations). This may be associated with superficial ion bombardment of growing layers ZrN_x by heavy ions Zr [21]. Herewith relaxation of deformation is difficult due to availability of closely located interphase boundaries.

Most universal characteristic of mechanical properties of coatings is their hardness.

In Figure 4 are showing data of hardness (*H*) and elasticity modulus (*E*) of multilayer coatings TiN_x/ZrN_x depending upon the number layers for two potentials of bias. From these figures understood that at the same number of layers (e.g. 100 layers) hardness at less constant potential (– 140 V) is equal to 42 GPa, and at $U_b = – 200$ V hardness is slightly lower and is 35 GPa. This indicates that at a greater constant potential is the maximum impact of radiation factor. Action of radiation factor leads to disordering and lower of hardness [15, 25]. We also note raise of hardness with increasing the number of periods both for $U_b = – 200$ V as well as $U_b = – 140$ V. The cause of identified effect may be an increase of specific density of interphase boundaries by increasing the number of layers. Interphase boundaries are preventing to displacement of dislocations and plastic discharge of deformation. Thus are increasing hardness and strength of material.

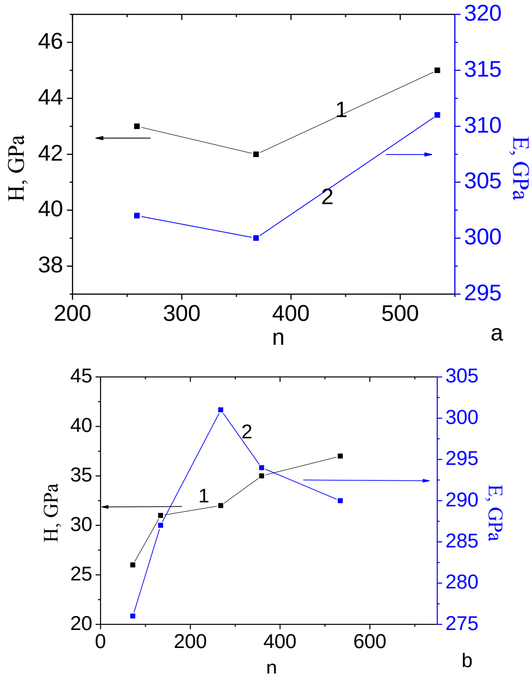


Fig. 4 – Graph of dependence of hardness (1) and elasticity modulus (2) of number of layers for two potentials of bias: – 140 V (a) and – 200 V (b)

Characteristic graph of change friction force for the pair "multi-period covering TiN_x/ZrN_x – diamond" depending on number of revolutions (N_r) is showed in Figure 5. It is seen that already after 5 revolutions (i.e. at the initial moment of tests) the dependence goes to values that are close to constant.

Low value of the friction coefficient is observed for compositional multilayer coatings TiN_x/ZrN_x ($f = 0.1$-0.14). Despite the fact that the hardness of these coatings are not highest among the investigated compositional pairs [21, 22].

Analysis of obtained results is showing that for pair « diamond - multiperiod coating TiN_x/ZrN_x" there is a dependence of friction coefficient from ratio *H*/*E*. Ratio *H*/*E* reflects the characteristic of resiliency of material.

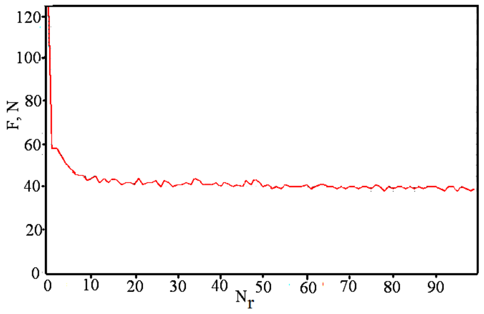


Fig. 5 – The curve of friction force change (*F*) for the pair "diamond - multiperiod coating TiN_x/ZrN_x" depending on number of revolutions (N_r), which determines friction path)

The conducted investigations of immersion depth of indenter in the pair "diamond – multiperiod coating TiN_x/ZrN_x" (Fig. 6) are proving that immersion depth of diamond indenter in the coating is also correlated with ratio *H*/*E*.

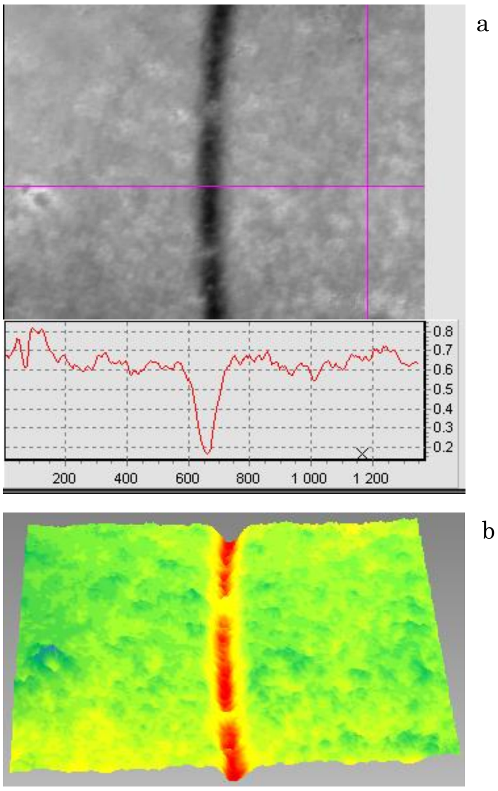


Fig. 6 – Images of surface multiperiod coating TiN_x/ZrN_x in the pair with diamond ($U_b = – 200$ V, $n = 268$): a – profilogram of coating surface, b – three-dimensional image of friction groove

Dependence of change of the friction coefficient f and introduction depths of indenter h from ratio *H*/*E* is showed in Fig. 7.

It is seen that with increase of the ratio *H*/*E* (i.e. at decreases of plasticity material and increasing of its relative hardness) is happening a decrease of penetration depth of indenter and herewith diminishing of the friction coefficient.

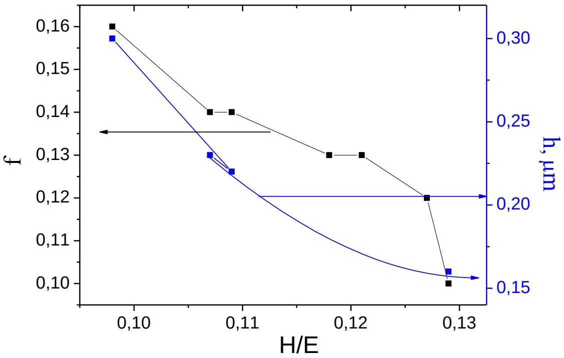


Fig. 7 – Dependence of the friction coefficient change f and penetration depth of indenter h from the ratio *H*/*E*

4. CONCLUSION

Thus, as used in the deposition conditions provide good planarity multiperiod structures TiN_x/ZrN_x.

Biphasic (TiN_x and ZrN_x) coatings condition that revealed by X-ray methods indicates about low specific contribution of section borders with mixed state in the phase composition and structure. Also this is showing low level of mixing for all used compositions with number of layers from 134 to 534.

On the substructure level most sensitive to U_b is micro-strain. Its growth with increasing the number of ZrN_x layers (at the greatest $U_b = -200$ V) testifies about defining contribution of defect formation that stimulated by irradiation of heavy Zr ions at coating formation.

It has been established that at optimal technological parameters of obtaining multiperiod nitride coatings TiN_x/ZrN_x their hardness is in the range 40-50 GPa that corresponds to superhard state.

Low value $f = 0.1$-0.14 was found at research of characteristic friction by method of introduction of indenter with rotation in the pair "diamond – multiperiod coating TiN_x/ZrN_x". Dependence of penetration depth of indenter and friction coefficient from ratio *H*/*E* that characterizes elasticity of the material was revealed.

Структура і фізико-механічні властивості многоперіодной вакуумно-дугових покриттів на основі двошарової системи TiN_x/ZrN_x

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Проведені комплексні дослідження методами структурного аналізу (прецизійний XRD-метод і растрова електронна мікроскопія) в поєднанні з випробуваннями на фізико-механічні властивості (твердість, модуль пружності, сила тертя і коефіцієнт тертя). Такі комплексні дослідження є основою для оптимізації властивостей многоперіодной систем TiN_x/ZrN_x шляхом зміни їх структурних станів (структурна інженерія). Основними параметрами зміни були: число шарів (*n*) від 134 до 534 (при загальній товщині покриттів близько 10 мкм) і величина негативного потенціалу зсуву U_b. Виявлено формування двофазного (TiN_x і ZrN_x) стану. На субструктурного рівні найбільш чутливою до U_b є мікродеформацій. Зростання мікродеформації зі збільшенням числа ZrN_x шарів (при найбільшому $U_b = -200$ В) свідчить про визначальний внесок опромінення важкими Zr іонами в утворення дефектів при формуванні покриття. Встановлено, що при оптимальних технологічних параметрах отримання многоперіодной нітридних покриттів TiN_x/ZrN_x їх твердість знаходиться в межах 40-50 ГПа, що відповідає надтвердому стану. Виявлено залежність глибини проникнення індентора при випробуваннях в парі «алмаз - багатошарове покриття $TiN_x/ZrN_{\text{Шх}}$» і коефіцієнта тертя від співвідношення *H*/*E*, яке характеризує пружність матеріалу.

Ключові слова: Багатошарове покриття, Число шарів, TiN_x/ZrN_x, Субструктура, Мікродеформація, Розмір кристалітів, Твердість, Глибина впровадження, Коефіцієнт тертя.

Структура и физико-механические свойства многопериодных вакуумно-дуговых покрытий на основе двухслойной системы TiN_x/ZrN_x

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Проведены комплексные исследования методами структурного анализа (прецизионный XRD-метод и растровая электронная микроскопия) в сочетании с испытаниями на физико-механические характеристики (твердость, модуль упругости, сила трения и коэффициент трения). Такие комплексные исследования являются основой для оптимизации свойств многопериодных систем TiN_x/ZrN_x путем изменения их структурных состояний (структурная инженерия). Основными параметрами изменения были: число слоев (n) от 134 до 534 (при общей толщине покрытий около 10 мкм) и величина отрицательного потенциала смещения U_b. Выявлено формирование двухфазного (TiN_x и ZrN_x) состояния. На субструктурном уровне наиболее чувствительной к U_b является микродеформация. Рост микродеформации с увеличением числа ZrN_x слоев (при наибольшем $U_b = -200$ В) свидетельствует об определяющем вкладе облучения тяжелыми Zr ионами в образовании дефектов при формировании покрытия. Установлено, что при оптимальных технологических параметрах получения многопериодных нитридных покрытий TiN_x/ZrN_x их твердость находится в пределах 40-50 ГПа, что соответствует сверхтвердому состоянию. Выявлена зависимость глубины проникновения индентора при испытаниях в паре паре «алмаз- многопериодное покрытие TiN_x/ZrN_x» и коэффициента трения от соотношения H/E, которое характеризует упругость материала.

Ключевые слова: Многопериодное покрытие, число слоев, TiN_x/ZrN_x, субструктура, микродеформация, размер кристаллитов, твердость, глубина внедрения, коэффициент трения.

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