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INFLUENCE OF PRESSURE OF WORKING ATMOSPHERE ON THE FORMATION OF PHASE-STRUCTURAL STATE AND PHYSICAL AND MECHANICAL PROPERTIES OF VACUUM-ARC MULTILAYER COATINGS ZrN/CrN

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For multilayer coating system ZrN/CrN determined the effect of the pressure of the working atmosphere of nitrogen (P_N), DC ($-U_s$) and pulse ($-U_i$) negative bias potential during the deposition and the thickness of the layers in the period on the phase composition, texture, substructural characteristics and physical-mechanical properties. It is found that for $P_N = (2.2...12)\cdot10^{-4}$ Torr in the layers of chromium nitride formed on a lower nitrogen phase with the β-Cr_2N simple hexagonal crystal lattice, and in the zirconium nitride layers are formed of a stoichiometric ZrN phase with a cubic lattice. Such a multilayer coating (layer thickness about 50 nm) at the maximum in this range $P_N = 1.2\cdot10^{-3}$ Torr is most solid (39 GPa) with a modulus of elasticity of 268 GPa and the ratio H/E = 0.145. At higher P_N, when the layers are formed phase stoichiometric composition with homogeneous crystal lattices (ZrN and CrN) hardness of the composition is not more than 33 GPa. The mechanisms of the effects observed are based on the higher barrier properties of the interphase boundary layers with different types of crystal lattices was discussed.

INTRODUCTION

Creating resistant to external influences wear resistant nitride coatings is the subject of many works of leading research groups in the field of surface engineering [1–14]. By the breakthrough achievements in this area in recent years can be attributed creation of the theory and practical obtaining materials multi-elemention composition and nitrides on their basis of a stable structure, and properties at high temperatures [15–17], as well as the development of the method of the structurtural engineering in the field of multi-layer coatings consisting of layers of different composition with certain (necessary technology) functionalities. In the latter case the highest functional characteristics, and particularly the physical and mechanical, have been achieved when the thickness of the layers lying in the nanometer range [18–33].

The establishment of such multi-layered synthetic materials makes it possible to carry out a wide range of structural engineering (and thus change the properties) using as a multilayer composition of different composition and properties of layers, and multiperiod of the periodic (mostly bi- and three-layers in the period) systems. At the present time among the little-known but promising in its potential capabilities of the system applies ZrN/CrN. In this system, the components are combined such as the ZrN, having high hardness, resistance to radiation and resistant to thermal stresses and also having sufficiently high hardness and wear resistance CrN.

In this pair CrN has most relevant concentration range of 30…50 at.% N two major structural modification (Fig. 1) [34], and it makes efficient structural engineering by different nitrogen saturation of layers, is achieved by changing the operating pressure nitrogen atmosphere during the deposition. Therefore, the aim of this work was to establish the patterns to the effect of the pressure of the working atmosphere on the phase-structural state, substructure and hardness of multiperiod ZrN/CrN coatings with different thickness and number of layers.

METHOD OF PRODUCTION AND RESEARCH SAMPLES

Multilayered two-phase nanostructures coatings ZrN/CrN deposited in vacuum-arc installing "Bulat-6" [35]. As the cathode materials used: low-alloyed chromium and zirconium; active gas – nitrogen (99.95%). The coatings were applied to the surface of samples 20x20x2 mm steel 12X18H10T prepared by standard methods of grinding and polishing. The procedure for the deposition of multilayer coatings includes the following operations. The vacuum chamber pumped out to a pressure of 10^{-5} Torr. Then, the rotator is supplied with negative potential of the substrate holder 1 kV switch on evaporator and the surface is cleaned first of the two substrates chromium ion bombardment for 3...5 min. After that, the substrate holder is rotated 180°, and performed the same treatment of the second substrate. Further switch on concurrently both evaporators, nitrogen was supplied to the chamber, and the deposition first layer ZrN one hand, and with anti-oppositely - CrN. Coatings were obtained as in the continuous rotation of the holder at 8 rev/min and the number of layers 540…570, and by a fixed stop on time 10, 20, 40 or 150 s at each of the 2 cathodes for thicker layers. The deposition was carried out under the following process conditions: arc current during the deposition was 100 A, nitrogen pressure (P_N) in the chamber was varied in the range of $5\cdot10^{-5}...10^{-3}$ Torr, the distance from the evaporator to the substrate – 250 mm, the substrate temperature (T_s) was in the range of 250...350 °C.

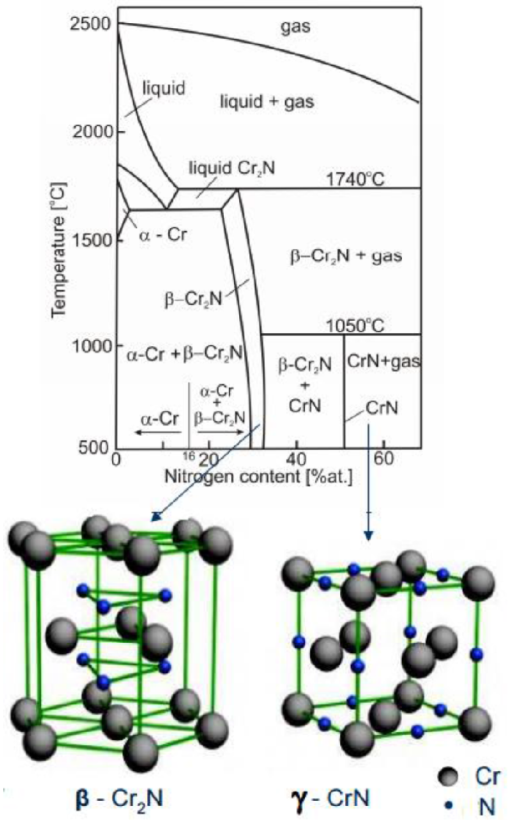

Fig. 1. The equilibrium diagram of the Cr-N and a schematic view of the cell of the crystal lattices 2 most relevant in forming coatings phases β-Cr_2N and γ-CrN

Obtained coating thickness of about 10 μm. Multilayer nanostructured coatings ZrN/CrN with growth stimulation was obtained by applying to substrate holder in the form of a high-voltage pulse potential with a pulse width of 10 μc, repetition rate of 7 kHz and an amplitude -800 V [36]. Phase composition, structure and substructural characteristics were studied by X-ray diffractometry (DRON-4) with using Cu-K_α-radiation. For monochromatization of detected radiation used graphite monochromator, which is installed in the secondary beam (front of the detector). The study of the phase composition, structure (texture, substructure) produced by means of traditional methods of ray diffractometry analysis position, intensity and shape profiles of diffraction reflections. To decrypt diffractograms was used patterns table International Centre for Diffraction Data Powder Diffraction File. Substructural characteristics were determined by approximation [37]. Microindentation performed on the "Micron-gamma" installation with a load up to F = 0,5N by Berkovich diamond pyramid with an angle of 65°, to automatically perform loading and unloading for 30 s.

RESULTS AND DISCUSSIONS

Analysis of the morphology obtained coatings on the results of scanning electron microscopy studies show high planarity and good homogeneity the coatings formed as a supplying constant negative bias potential ($-U_s$), both with and without filing $-U_s$, but using in this case for stimulating mobility of deposited atoms supplying high voltage potential ($-U_i$ = -800 V) in the form of a pulse (Fig. 2).

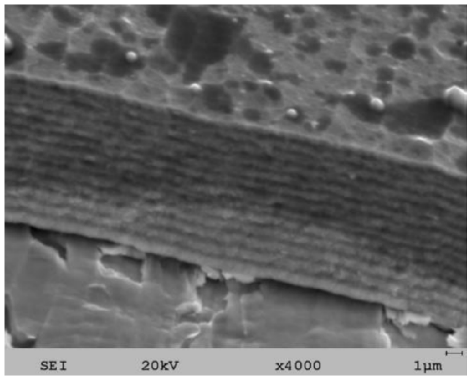

Fig. 2. The morphology of the multilayer coating side chipped ZrN/CrN with 24 layers

Corresponding coatings, that X-ray diffraction spectra show that when the pressure is reduced during the deposition of $4\cdot10^{-3}$ to $1.5\cdot10^{-3}$ Torr as in the ZrN layer, and the CrN layer is formed crystallites with preferred orientation axis [100] perpendicular to the plane of growth (Fig. 3) significantly increased the relative intensity of the reflections (200). The formation of such orientation in the deposition vacuum arc nitride coatings with the structural type NaCl of crystal lattice is usually associated with the depletion of the condensate of light nitrogen atoms [14, 37, 38]. As have shown results testing the hardness, the formation of such texture resulting in increased of material hardness of 30 to 36 GPa.

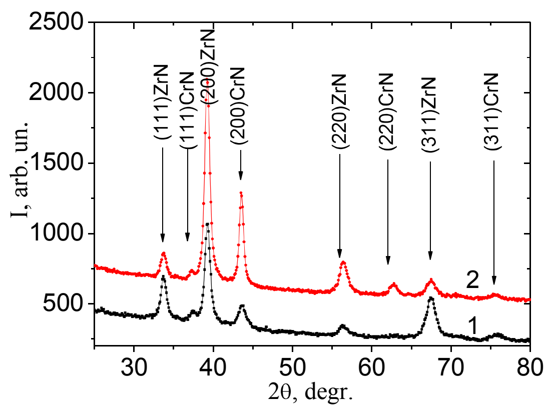

Fig. 3. Plots of diffraction spectra coatings ZrN/CrN, obtained by pulsed high-voltage-potential ($-U_i$ =-800V) and working pressure nitrogen atmosphere P_N, Torr: 1 – $4\cdot10^{-3}$; 2 – $1.5\cdot10^{-3}$

Filing a constant negative bias potential $-U_s$ = -70 V at a pressure of $P_N = 4\cdot10^{-3}$ Torr is accompanied by the appearance of the texture with the axis [111]. In this case, when pressure is decreased from $4\cdot10^{-3}$ to $6.2\cdot10^{-4}$ Torr occur structural phase changes in layers: in Zr-N – appears texture [100], and Cr-N – shaped lower nitride Cr_2N (Fig. 4,a). Wherein the hardness decreases from 28 to 20 GPa. Feed simultaneously with constant and high voltage potential magnitude $-U_i$ = -800 V in pulse form does not lead to qualitative differences in the type of generated at different pressures phases, nor characteristic of preferred orientation: with [100] axis at a low pressure, and [111] – when a large P_N (see Fig. 4,b). However, the hardness such coatings is somewhat higher than without feed $-U_i$ and changes from 30 to 33 GPa. Using during the deposition more constant bias potential $-U_s$ = -150 V accompanied by the appearance of the lower nitrogen phase (Fig. 5), not only at relatively low (2...6)·10^{-4} Torr, but at a higher pressure (i. e., high pressures manifest non-stoichiometry). At low pressure P_N = 2.2·10^{-4} Torr layers based on chromium during the deposition nitride phase is not formed (spectrum 4 in Fig. 5,a). The hardness of such composite coatings thus decreases from 39 GPa at the maximum pressure up to 21 GPa at a pressure of P_N = 2.2·10^{-4} Torr. Use in this case, the second type of impact potential high voltage magnitude -U_i = -800 V in pulse form is also fundamentally does not changes the phase composition of the coatings but changes the orientation of crystallites. In the greatest measure this affects at a pressure of 6.2·10^{-4} Torr, stimulating the formation of texture [111] β-Cr_2N. wherein the hardness is increasing of 30 GPa (without -U_i) and 32 GPa (feeding -U_i).

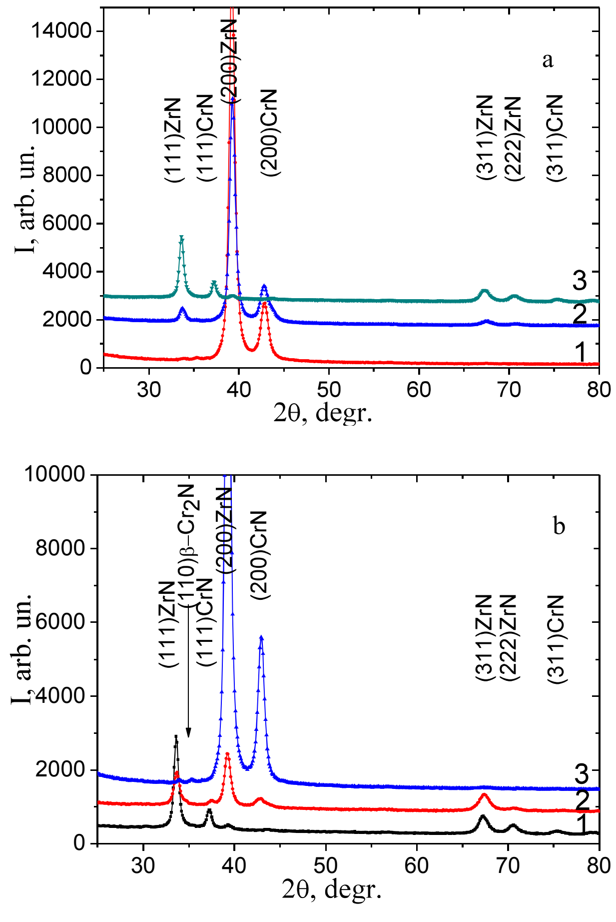

Fig. 4. Plots of diffraction spectra of ZrN/CrN coatings with thickness of layers about 50 nm obtained by -U_s = - 70 V and P_N, Torr: 1 – 4·10^{-3}; 2 – 1.2·10^{-3}; 3 – 6,2·10^{-4}; a – at -U_i = 0; b – at -U_i = -800 V

Researches on the substructure level which conducted by approximation method showed that an increase in pressure in the coatings deposited at -U_s = -70 V is increased crystallite size with decreasing of microstrain (Fig. 6,a). Submission of additional high voltage potential -U_i = -800 V in the form of a pulse does not lead to a fundamental change in the course of dependency, but significantly reduces the average crystallite size and microstrain value.

The increase in supply during the deposition of a constant potential -U_s to a value of -150 V accompanied by a relative increase in the L in the direction of incidence of film-forming particles (≈ 15...20%). It is also an explicit effect is the relative decline in microstrain at low pressure. Feeding at -U_s = -150 V action additional potential in the form of pulse (-U_i = -800 V) also significantly reduces L by 20...25% and a decrease in <ε> almost 2 times (to 0.3%) with low pressure deposition.

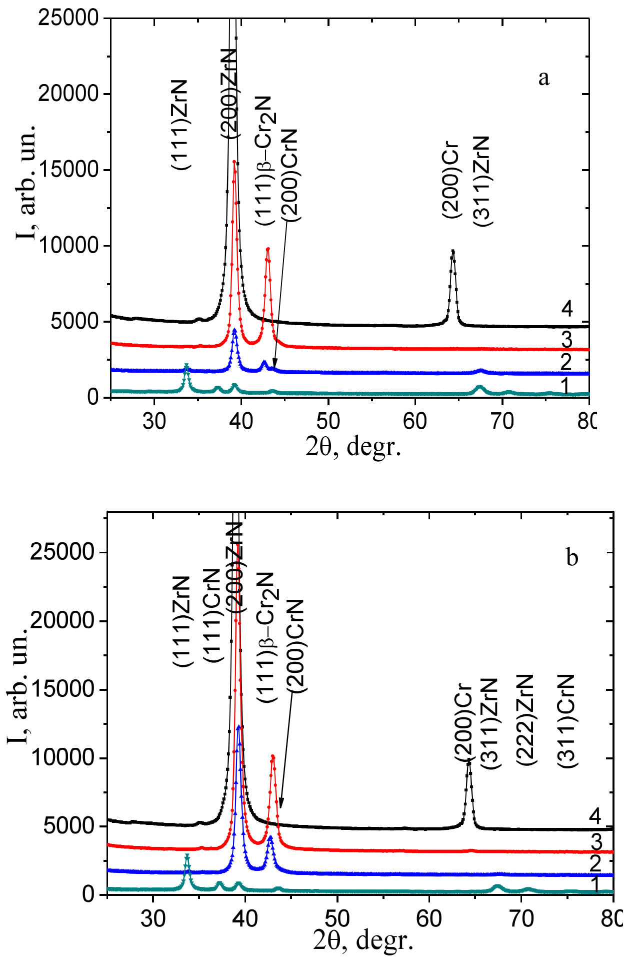

Fig. 5. Plots of diffraction spectra of ZrN/CrN coatings with thickness of layers about 50 nm obtained by -U_s = -150 V and P_N, Torr: 1 – 4·10^{-3}, 2 – 1.2·10^{-3}, 3 – 6.2·10^{-4}, 4 – 2.2·10^{-4}; a – at -U_i = 0, b – at -U_i = -800 V

It should be noted that the substructural characteristics are largely changed by varying the layer thickness. Thus, when -U_s = -150 V and a pressure P_N = 4·10^{-3} Torr with increasing average layer thickness of 25 to 220 nm is not only to the expected increase L proportional thickness of the layers, but in the ZrN layer increases <ε>, and in layer CrN – occurs its decrease (see Fig. 6,c).

Submission of additional potential -U_i = -800 V practically does not change the absolute values and the dependence on the thickness of <ε> (h) and L (h), whereas the supply is only -U_i = -800 V (with -U_s = = 0 V) leads to a significant increase in <ε> and L decrease (see Fig. 6,d).

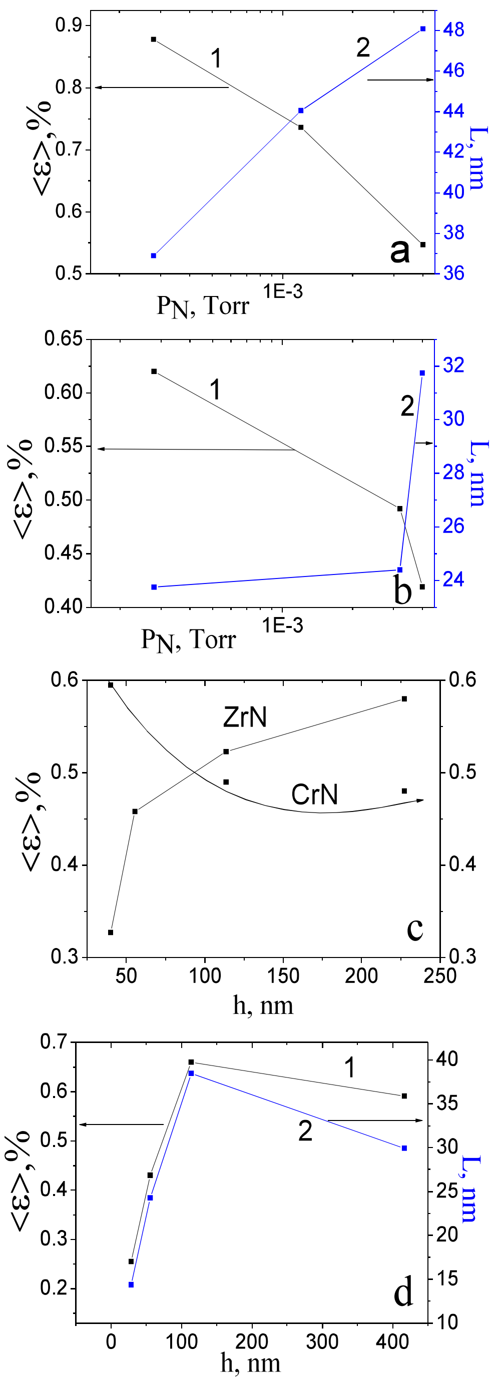

Fig. 6. Dependence substructural characteristics (microstrain <ε> and the crystallite size L) in ZrN layers of pressure (a, b) to ZrN layer thickness (d) and the thickness of the layers in ZrN and CrN (c) coating obtained by:

a – $-U_s$ = -70 V and $-U_i$ = 0 V;

b – $-U_s$ = -70 V and $-U_i$ = -800 V;

c – $-U_s$ = -150 V and $-U_i$ = 0 V;

d – $-U_s$ = 0 V and $-U_i$ = -800 V

Comparison of $-U_s$ influence on substructural characteristics ZrN and CrN layers shows that the decrease in the magnitude of supply negative constant bias potential of -150 to -70 V leads to increase <ε> and L in ZrN layers and decrease in the average values of L and <ε> in layers of CrN. With increasing layer thickness microstrain decreases in CrN layers, and in ZrN layers – increases.

A sharp drop in the level of <ε> at the lowest layer thickness of 25...50 nm due to the fact that the magnitude of the crystallites L on the nanoscale their formation is proportional to the thickness of the layers, and at achievement its smallest size 20...30 nm makes it impossible to work in the crystallites dislocation source Frank-Reed and formation wherein the random dislocation of reset strain [39].

To study the influence of the deposition parameters on the physical and mechanical properties of composite coatings have been used in such universal and express criteria such as hardness and elastic modulus, defined according to the data of dynamic microindentation [40]. Previously, when analyzing the effect of pressure on the phase composition it has been shown that the formation of a layer of chromium-based lower β-Cr_2N nitride at a pressure $P_N = (2.2...6.2)\cdot 10^{-4}$ Torr leads to a substantial drop in hardness. Constructed for the entire operating investigated range of the $P_N = (2.2...40)\cdot 10^{-4}$ Torr generalized dependence H (P_N) shown in Fig. 7,a shows that the coatings deposited at $-U_s$ = -150 V a observed nonmonotonic dependence of the microhardness on the working pressure nitrogen atmosphere with a maximum of H = 39 GPa in the $P_N = 1.2\cdot 10^{-3}$ Torr. In case of submission $-U_i$ = -800 V depending H (P_N) becomes smoother (see curve 2 in Fig. 7,a), which may be associated with greater planar mobility of additional acceleration in pulsed potential film-forming charged particles, which leads to greater planarity growing layers and the sharp border of the interlayer.

Similar behavior depending on H (P_N) can be traced to $-U_s$ = -70 V, but the absolute value obtained in this case, the microhardness does not exceed 33 GPa.

CONCLUSIONS

In work established pressure range $P_N = (2.2...12)\cdot 10^{-4}$ Torr in which in the layers Zr-N and Cr-N phase formed with different types of lattices: in a layer of zirconium nitride – ZrN phase with a cubic an FCC lattice and a layer of chromium nitride – β-Cr_2N phase hexagonal crystal lattice. In this range, with increasing pressure P_N is formed predominantly oriented crystallites, which leads to increasing the hardness to 39 GPa.

At higher $P_N = 4\cdot 10^{-3}$ Torr formation in both layers of stoichiometric and thus the same by type of crystal lattices ZrN and CrN phase (cubic crystal lattice structural type NaCl) is accompanied by a decrease in hardness of multiperiod coating of 30…33 to 39 GPa. Such effect is defined smaller permeability of dislocations and higher diffusion stability boundaries phases with different crystal lattices of the type that defines a higher barrier properties and indentation accordingly leads to higher hardness of the composition.

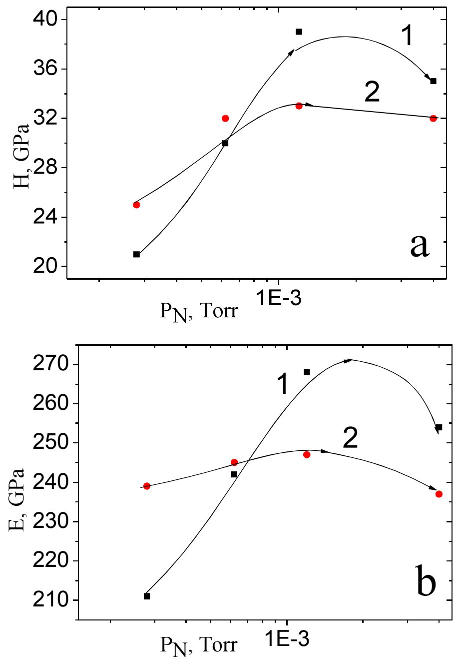

Fig. 7. Effect of pressure in the deposition of coatings on their hardness (a) and modulus of elasticity (b): 1 – U_s = - 150 V; 2 – -U_s = - 70 V

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ВЛИЯНИЕ ДАВЛЕНИЯ РАБОЧЕЙ АТМОСФЕРЫ НА ФОРМИРОВАНИЕ ФАЗОВО-СТРУКТУРНОГО СОСТОЯНИЯ И ФИЗИКО-МЕХАНИЧЕСКИЕ СВОЙСТВА ВАКУУМНО-ДУГОВЫХ МНОГОСЛОЙНЫХ ПОКРЫТИЙ ZrN/CrN

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Для многослойных покрытий системы ZrN/CrN определены влияния давления рабочей азотной атмосферы (P_N), постоянного ($-U_s$) и импульсного ($-U_i$) отрицательных потенциалов смещения при осаждении, а также толщины слоев в периоде на фазовый состав, текстуру, субструктурные характеристики и физико-механические свойства. Установлено, что при $P_N = (2,2…12)\cdot10^{-4}$ Торр в слоях нитрида хрома формируется низшая по азоту фаза β-Cr_2N с простой гексагональной кристаллической решеткой, а в слоях нитрида циркония происходит формирование стехиометрической фазы ZrN с кубической решеткой. Такое многослойное покрытие (толщина слоев около 50 нм) при наибольшем из этого интервала $P_N = 1,2\cdot10^{-3}$ Торр является наиболее твердым (39 ГПа) с модулем упругости 268 ГПа и отношением H/E = 0,145. В случае более высоких P_N, когда в слоях происходит образование фаз стехиометрического состава с однотипными кристаллическими решетками (ZrN и CrN), твердость композиции не превышает 33 ГПа. Обсуждены механизмы наблюдаемого эффекта, основанные на более высоких барьерных свойствах межфазной границы слоев с различными типами кристаллических решеток.

ВПЛИВ ТИСКУ РОБОЧОЇ АТМОСФЕРИ НА ФОРМУВАННЯ ФАЗОВО-СТРУКТУРНОГО СТАНУ І ФІЗИКО-МЕХАНІЧНІ ВЛАСТИВОСТІ ВАКУУМНО-ДУГОВИХ БАГАТОШАРОВИХ ПОКРИТТІВ ZrN/CrN

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Для багатошарових покриттів системи ZrN/CrN визначено вплив тиску робочої азотної атмосфери (P_N), постійного ($-U_s$) і імпульсного ($-U_i$) негативних потенціалів зсуву при осадженні, а також товщини шарів у періоді на фазовий склад, текстуру, субструктурні характеристики і фізико-механічні властивості. Встановлено, що при $P_N = (2,2...12)\cdot10^{-4}$ Торр у шарах нітриду хрому формується нижча по азоту фаза β-Cr_2N з простою гексагональною кристалічною решіткою, а в шарах нітриду цирконію відбувається формування стехіометричної фази ZrN з кубічною решіткою. Таке багатошарове покриття (товщина шарів близько 50 нм) при найбільшому з цього інтервалу $P_N = 1,2\cdot10^{-3}$ Торр є найбільш твердим (39 ГПа) з модулем пружності 268 ГПа і співвідношенням H/E = 0,145. У випадку більш високих P_N, коли в шарах відбувається утворення фаз стехіометричного складу з однотипними кристалічними решітками (ZrN і CrN), твердість композиції не перевищує 33 ГПа. Обговорено механізми спостережуваного ефекту, що засновані на більш високих бар'єрних властивостях міжфазної межі шарів з різними типами кристалічних решіток.