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REGULAR ARTICLE

Influence of Shielding Potential on the Formation Process of Nanostructured Nitride Coatings of the MoN/CrN System

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Using the method of vacuum-arc deposition, multilayer MoN/CrN nitride coatings were synthesized under two different substrate bias potentials, specifically $U_b = -100$ V and $U_b = -200$ V. These coatings were deposited at a working nitrogen pressure of $P_N = 0.53$ Pa. The structural and mechanical properties of the obtained coatings were systematically studied, with a particular focus on microhardness and thermal stability. The investigation revealed that increasing the bias potential from – 100 V to – 200 V led to a significant enhancement in the hardness of the coatings. Specifically, the coatings deposited at $U_b = -200$ V demonstrated a 23.5 % higher hardness compared to those produced at $U_b = -100$ V, indicating improved densification and possibly finer microstructural features induced by the higher energy ion bombardment. The measured microhardness values ($HV_{0.05}$) of the as-deposited coatings were 25.6 GPa for $U_b = -100$ V and 28.8 GPa for $U_b = -200$ V. These values confirm the beneficial effect of a higher negative substrate bias on the mechanical performance of the multilayer architecture. Furthermore, post-deposition annealing at $T = 700$ °C was performed to assess the thermal stability and possible phase transformations. Interestingly, annealing did not result in an increase in hardness; on the contrary, a slight reduction in average microhardness was observed for the coating deposited at $U_b = -100$ V, with the value decreasing to 24.3 GPa. However, for the coating obtained at $U_b = -200$ V, annealing led to a slight increase in hardness, reaching 30.8 GPa, suggesting enhanced thermal stability and resistance to softening. These findings highlight the importance of deposition parameters in tailoring the performance of multilayer nitride coatings for high-temperature and wear-resistant applications.

Keywords: Vacuum-arc deposition, Multilayer coatings, Hardness, Adhesive strength.

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1. INTRODUCTION

To improve the functional properties, it is effective to move to multilayer compositions, which use alternating layers of coatings with different compositions. [1-3]. Due to the peculiarities of their structure, a significant number of layers and their thickness in the nanometer range, coatings with a submicrocrystalline structure and nanostructured coatings combine the advantages of layered systems and the specific properties of nanoscale objects. This makes them extremely effective for use in conditions of intense mechanical, thermal and chemical stress.

Ion-plasma deposition methods, such as magnetron sputtering and vacuum arc deposition, are the most widely used in industry to produce protective coatings.

To understand the mechanism of formation of multilayer coatings, it is necessary that the layers have different shear moduli and, consequently, different dislocation line energies, and the thickness of the layers must be thin enough to limit the formation and movement of dislocations in individual layers. In this case, two levels of modulation are required, which should form consistent interfaces. For a multilayer coating that meets these criteria, a significant internal stress is required to move the dislocation along the interface, and, therefore, to increase the hardness of the entire coating Development of new protective coating materials, methods and processes for their application to create multilayer structures with improved performance characteristics compared to monolayer structures that meet modern requirements for surface protection of cutting tools operating at elevated temperatures, fatigue and thermal fatigue loads.

The issue of protecting the surface of cutting tools in elevated temperature conditions, when the temperature in the cutting zone can reach 800 – 900 °C, is particularly acute. A promising area is the use of multilayer coatings consisting of periodically arranged layers of different materials of nanometer thickness. Such coatings

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are characterized by high mechanical and operational properties. Due to the specific structure, large number of layers and their thickness in the nanometer range, nanostructured coatings demonstrate improved performance.

The greatest effect is achieved with nanometer-sized layers, since it is in this range that nitrides exhibit the highest mechanical properties. Ion-plasma technologies, in particular the vacuum-arc deposition method, are probably associated with the bombardment of energetic ions during the deposition of layers under the condition of a negative stress shift on the substrate. This contributes to the compaction of interlayer boundaries and an increase in residual stresses, which, in turn, provides an increase in hardness compared to the original materials.

Combining high-hardness layers into a single multilayer coating allows high hardness to be maintained by forming a large number of strong interfaces, which significantly increases the overall strength of the material.

One of the most promising materials for multilayer coatings is chromium nitride (CrN). This material exhibits high wear resistance, corrosion resistance, and stability at high temperatures, making it ideal for protecting cutting tools that operate at high cutting speeds. Also, chromium nitride has a lower coefficient of friction compared to titanium nitride (TiN), which is widely used in industry. However, single-layer coatings made of chromium nitride have certain limitations: they are not hard enough and are prone to rapid abrasive wear.

To solve these problems, it is necessary to improve the structure of the coating by creating multilayer architectures. In such systems, chromium nitride is combined with other materials that complement its properties. For example, the use of molybdenum nitride (MoN) as a second component is effective. Molybdenum nitride is known for its high mechanical properties, which provide additional strengthening of the coating.

MoN/CrN multilayer coatings combine the advantages of both materials: CrN provides high wear resistance and low friction coefficient, while MoN gives the coating additional hardness and increased resistance to thermal and mechanical stress. The interaction between the layers of these materials contributes to the formation of a large number of strong interfaces, which increases the overall strength and performance of the coating.

The development of new protective coating materials, methods and processes for their application to create multilayer structures with improved performance characteristics compared to monolayer structures that meet modern requirements for protecting the surface of cutting tools operating at elevated temperatures, fatigue and thermal fatigue loads is an urgent task of modern engineering.

In [4], the effect of chromium (Cr) on the structural evolution and mechanical properties of Mo-N coatings obtained at different partial nitrogen pressures was studied. The coatings synthesized at (P_{N2}/P_T) ratios of 0.32 and 0.44 show a structural organization that is best described by the quasi-binary Mo_2N-CrN bonding line. Mo_2N and CrN have a face-centered cubic (FCC) structure, with half of the nitrogen sublattice being vacant for Mo_2N.

With increasing Cr content, the vacancy of the nitrogen sublattice decreases. The chemical composition of the single-phase ternary component formed by the HCC is described as $Mo_{1-x}Cr_xN_{0.5(1+x)}$. The coatings demonstrate the agreement between the experimentally and computationally (ab initio) obtained lattice parameters of the $Mo_{1-x}Cr_xN_{0.5(1+x)}$ HCC.

Thus, the effect of Mo content up to 30.4 at. % on the microstructure and mechanical properties of CrN coatings was investigated in [5]. The ternary Cr-Mo-N coatings were deposited on steel substrates (AISI D2) using a hybrid method of arc ion deposition (AIP) using a Cr target and a DC magnetron sputtering method using a Mo target in a gaseous N_2/Ar mixture. The synthesized Cr-Mo-N coatings formed a solid substitution solution of (Cr, Mo)N, where larger Mo atoms replaced Cr in the CrN crystal. The Cr-Mo-N coatings showed an increased hardness value of approximately 34 GPa at 21 at. % Mo compared to 18 GPa for pure CrN. The coefficient of friction decreased from 0.49 for the pure CrN coating to 0.37 for Cr-Mo-N with 30.4 at. % Mo. It is believed that this result is due to the formation of a MoO_3 tribo-layer, which is known to function as a solid lubricant investigates the effect of molybdenum (Mo content up to 30.4 at. %) on the microstructure and mechanical properties of CrN coatings. Hardness and toughness were considered as the main parameters for improving the protective properties of coatings. Multilayer coatings with different bilayer periods, from 40 nm to 2.2 μm, were obtained by cathodic arc physical vapor deposition (Arc-PVD) on a stainless steel substrate.

Multilayer coatings with different bilayer periods, from 40 nm to 2.2 μm, were obtained by cathodic arc physical vapor deposition (Arc-PVD) on a stainless steel substrate. The hardness and toughness were studied as the main parameters for improving the protective properties of the coatings [6]. The mechanical properties of the deposited films were studied by measuring the hardness (H) and Young's modulus (E) using microindentation, and the H/E and H_3/E_2 ratios were calculated. The dependence of the internal structure and, consequently, the mechanical properties on the thickness of the film layer was revealed. A significant increase in hardness and toughness was observed with a decrease in the thickness of a single layer to 20 nm: $H = 38 - 42$ GPa, $H/E = 0.11$.

In [7], the mechanical and thermal properties of multilayer TiN/CrN_M1 and _M2 coatings with bilayer periods of ~17.8 and 6.7 nm were studied. The interfacial strengthening resulting from the coherent interface leads to an increase in hardness of ~27.5 GPa for TiN/CrN_M1 and ~29.4 GPa for TiN/CrN_M2, which corresponds to ~26.4 GPa for TiN and ~17.3 GPa for CrN. The inserted TiN layer slows down the thermal decomposition process of CrN due to the higher binding energy of Ti-N than Cr-N, and thus demonstrates the higher thermal stability of TiN/CrN multilayer coatings. In addition, TiN/CrN multilayer coatings show better oxidation8resistance than TiN coatings.

In [8, 9], two series of coatings were obtained by studying the influence of the main technological parameters on the deposition process (the value of the negative shear potential, the pressure of the reaction gas) (deposition parameters are given in Table 1).

Table 1 – Physical and technological parameters of MoN/CrN coating deposition

Series	P_N, mmHg	U_b, V
a	7×10^{-4}	– 20; – 70; – 150
b	3×10^{-3}	– 20; – 70; – 150; – 300

The highest hardness value of 38.0 GPa, which corresponds to the superhard state, is observed in the MoN/CrN coating at $P_N = 3 \times 10^{-3}$ mm Hg and $U_b = -70$ V. The maximum load of adhesive fracture of 145-159 N is inherent in MoN/CrN coatings obtained at the parameters: $P_N = 3 \times 10^{-3}$ mm Hg and $U_b = -70 - -150$ V.

The paper deals with the formation of MoN/CrN coatings based on refractory metal nitrides with nanometer layer thicknesses and the influence of physical and technological deposition parameters on their structural and phase state, mechanical and properties.

2. EXPERIMENTAL PART

To achieve this distance between the substrate and the evaporators, the focusing magnetic coils were removed, leaving only the stabilizing coils. The samples were polished substrates made of stainless steel 12X18N9T with dimensions of 18 × 18 × 2.5 mm.

The elemental composition of the samples was studied by analyzing the spectra of characteristic X-ray radiation generated by an electron beam in an electron microscope. The spectra were recorded using EDAX energy-dispersive X-ray spectrometers installed in scanning electron microscopes. The calculation of the element content in the studied material was carried out using the program supplied with the scanning electron microscope.

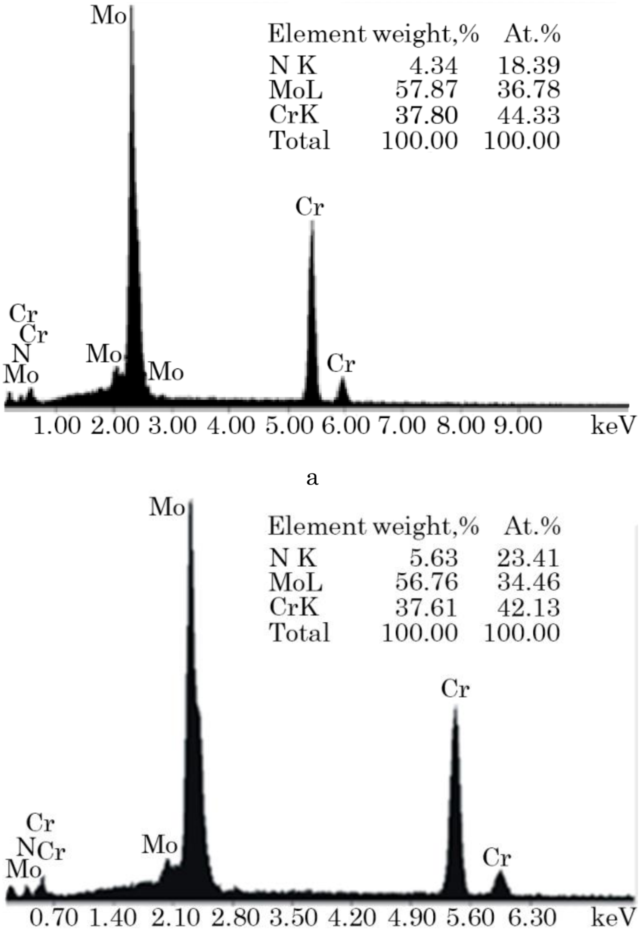
The study of the structural and phase state was carried out on a DRON-4 diffractometer in Cu-Kα radiation (wavelength = 0.154 nm) using a graphite monochromator in the secondary beam. The diffraction spectrum for phase analysis was recorded in the θ-2θ scanning scheme with Brague-Brentano focusing in the angle range $2\theta = 25 - 90°$. The recording was carried out in a point-by-point mode with a scanning step $\Delta(2\theta) = 0.02…0.2°$ and the duration of pulse accumulation at each point was 10…100 s (depending on the width and intensity of the diffraction maxima).

Microhardness measurements of coatings were performed using an automated AFFRI DM-8 hardness tester using the micro-Vickers method. Indentations were applied in series of 10 times for each load, at a distance of at least three diagonal lengths of the indentation from each other. The samples were fixed on a special holder and placed under the indenter. The place of application of the indentation was selected using a built-in optical microscope. The exposure time was 10 s. The determination of the Vickers hardness (HV) value was carried out in automatic mode. The movement of samples under the microscope, as well as under the indenter, was carried out using a motorized stage controlled by a computer.

A high-temperature vacuum furnace VHT 8/22-GR was used to anneal the coated samples. This furnace is equipped with a graphite chamber and molybdenum heating elements. Thanks to the ability to constantly change the conditions of the heating process and numerous additional equipment, complex heating can be performed in this furnace

3. RESULTS AND DISCUSSION

The results of chemical analysis of the coatings presented in Fig. 1.1 indicate a change in the concentration of elements in the coating composition with the bias potential to the substrate.



Element	weight,%	At.%
N K	4.34	18.39
MoL	57.87	36.78
CrK	37.80	44.33
Total	100.00	100.00

a

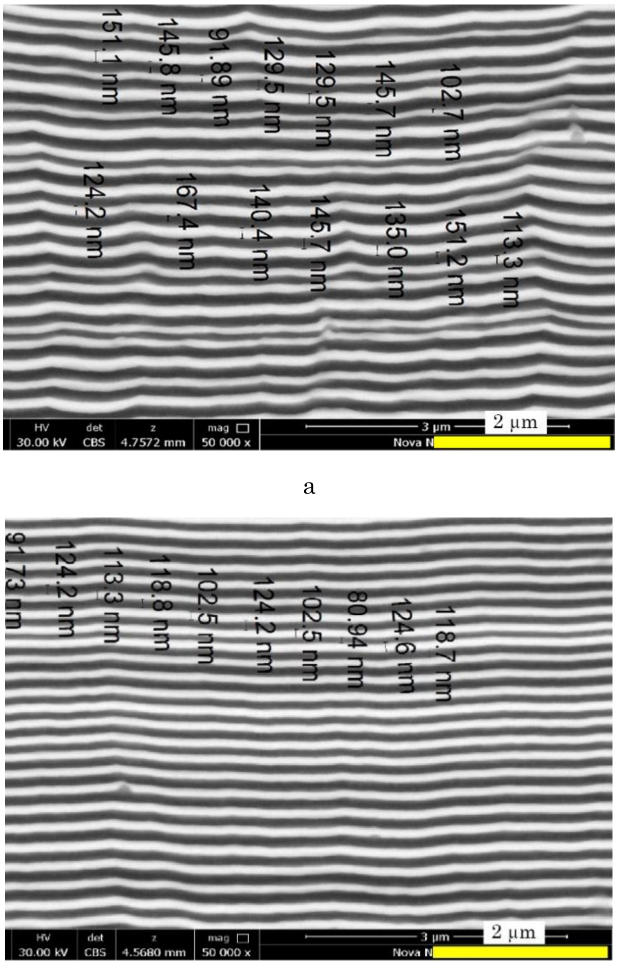
Element	weight,%	At.%
N K	5.63	23.41
MoL	56.76	34.46
CrK	37.61	42.13
Total	100.00	100.00

b

Fig. 1.1 – Energy spectra of the chemical composition of MoN/CrN multilayer coatings obtained at a nitrogen pressure of 0.53 Pa and a bias potential on the substrate of – 100 V (a) and – 200 V (b)

A detailed study of the nitrogen content of the coatings showed that the application of a bias potential significantly affects the change in the nitrogen (N) content of the coatings. This phenomenon is probably due to the secondary selective sputtering of light nitrogen atoms from the coating surface. With an increase in the modulus of the negative bias potential to – 200 V, an increase in the nitrogen content of the coatings is observed. At the same time, the amount of molybdenum and chromium in the coatings decreases. This change in elemental composition is the result of an increase in the efficiency of nitride compound formation, as higher values of the bias potential contribute to better binding of nitrogen atoms to metal elements, in particular molybdenum and chromium. This, in turn, leads to a change in the mechanisms of deposition and formation of nitride phases, which can positively affect the characteristics of coatings, such as hardness, wear resistance, and corrosion resistance [6].

Fig. 1.2 shows a high-resolution cross-sectional image of MoN/CrN multilayer coating samples, which clearly shows the characteristic wavy structure of the layers, which confirms the above theoretical justifications.

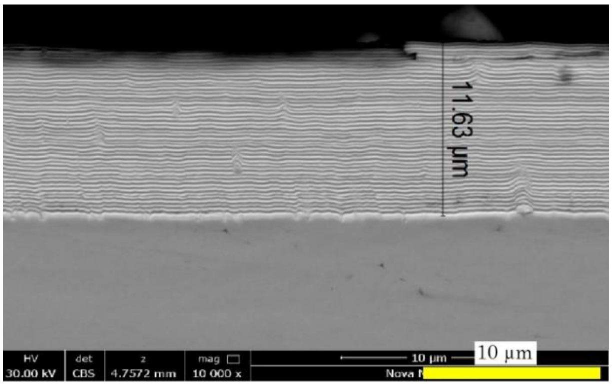


a

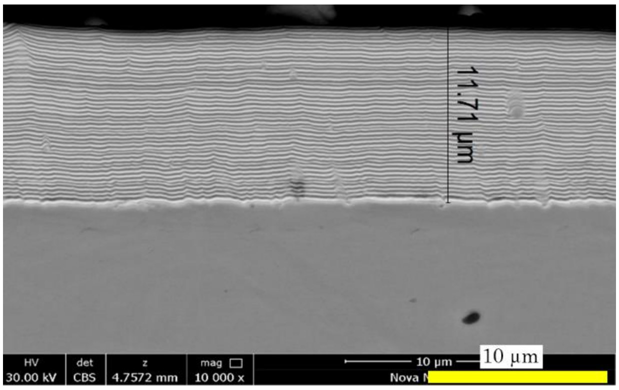
b

Fig. 1.2 – High-resolution micrographs of cross-sections of MoN/CrN multilayer coatings obtained at a nitrogen pressure of 0.53 Pa and a bias potential on the substrate of – 100 V (a) and – 200 V (b)

The analysis of microscopic cross-sectional images of MoN/CrN multilayer coatings shown in Fig. 1.2 shows that there is some unevenness in the thickness of the formed coating layers along the depth.



a



b

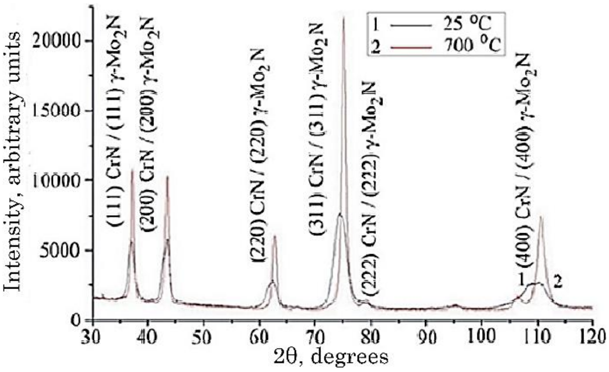
Fig. 1.3 – Micrographs of cross-sections of MoN/CrN multilayer coatings obtained at a nitrogen pressure of 0.53 Pa and a bias potential on the substrate of – 100 V (a) and – 200 V (b)

The total thickness of the coating (Fig. 1.3) averages approximately 11.6 μm. The inaccuracy is due to the thickness of the transition zone between the coating and the substrate and unevenness due to the droplet phase on the surface of the coatings. Black layers (see Fig. 1.2) are Mo$_2$N layers, and the light ones are CrN. The thickness of Mo$_2$N is slightly larger than that of CrN, which is due to the fact that the deposition rates of the materials are different (for chromium 3 nm/s, for molybdenum 4.5 nm/s).

The MoN/CrN multilayer coatings obtained at a nitrogen pressure of 0.53 Pa and a bias potential of – 100 V on the substrate had a bilayer thickness of $\lambda \sim 145$ nm, and at a nitrogen pressure of 0.53 Pa and a bias potential of – 200 V on the substrate, a bilayer thickness of $\lambda \sim 120$ nm.

The results of structural-phase studies of MoN/CrN multilayer coatings obtained at a nitrogen pressure of 0.53 Pa and bias potentials of – 100 V and – 200 V on the substrate are shown in Fig. 1.4 in the form of X-ray diffraction spectra. It was found that all the coatings are single-phase, with the (Cr, Mo)N phase (where chromium and molybdenum replace each other) being the main phase, since the peaks in the spectra cannot be clearly divided into peaks of two separate phases.

From the obtained diffraction spectra, it can be seen that phases with a low nitrogen content are formed in the coatings of both layers: in the MoN layer, it is the γ-Mo$_2$N phase with a cubic crystal lattice (structural type NaCl, JCPDS 25-1366), and in the layers of the CrN system, CrN with a cubic lattice (structural type NaCl, JCPDS 11-0065).



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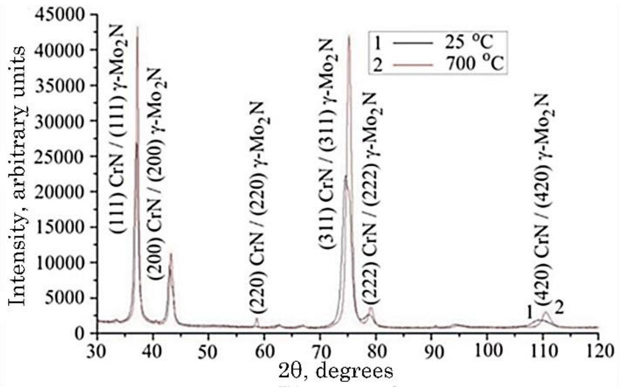

b

Fig. 1.4 – Sections of X-ray diffraction spectra of MoN/CrN multilayer coatings obtained at a nitrogen pressure of 0.53 Pa and a bias potential on the substrate of – 100 V (a) and – 200 V (b) before (black line 1) and after (red line 2) annealing

The γ-Mo$_2$N phase has a wide region of homogeneity, and cubic lattices are the most stable in non-equilibrium vacuum arc deposition methods [10]. Therefore, regardless of the increase in the bias potential on the substrate from – 100 V to – 200 V, γ-Mo$_2$N remains the main phase in the layers formed from MoN, and CrN remains the main phase in the layers formed from CrN.

For the multilayer MoN/CrN coating obtained at a substrate bias potential of – 200, the diffraction spectra begin to be dominated by peaks corresponding only to the isostructural phases (111)CrN/(111)γ-Mo$_2$N, indicating that the specific contribution of interfacial interaction in the layers decreases. The obtained results confirm that changes in the structure of coatings depend on the technological conditions of deposition, in particular, the bias potential on the substrate.

It is obvious that the intensity of the peaks in the XRD patterns of both coatings increased after annealing at 700 °C. Such changes in intensity usually indicate an increase in the number or better orientation of crystallites in the coatings, which can be caused by several factors: an increase in the number of phases, an increase in crystallinity, and an improvement in the orientation of crystallites.

Table 2 shows the lattice parameters of MoN/CrN multilayer coatings obtained at a nitrogen pressure of 0.53 Pa and a bias potential on the substrate of – 100 V (a) and – 200 V (b) before and after annealing at 700 °C.

Table 2 – Parameters of MoN/CrN multilayer coatings obtained at a nitrogen pressure of 0.53 Pa and a substrate bias potential of – 100 V and – 200 V before and after annealing at a temperature of 700 °C

T, °C	Phase	Lattice arameter, nm	U_b, V
25	γ-Mo$_2$N	0.4214	– 200
700	CrN	0.4188	
25	γ-Mo$_2$N	0.4209	– 100
700	CrN	0.4187	

It is obvious that the lattice parameters of coatings decrease during annealing, which indicates several possible phenomena related to the processes that occur during heat treatment. First of all, during annealing, when the temperature of the coating increased, thermal compression of the lattice was observed, which means a decrease in its parameters. This is due to the thermal expansion of individual atoms in the coating, which can result in a decrease in interatomic distances in certain phases of the coating. Additionally, atomic diffusion can occur during annealing, which can lead to changes in the composition and structure of the material. This can be particularly important for experimental MoN/CrN coatings, where different atoms can interact and change their lattice arrangement, resulting in changes in lattice parameters.

In addition, the annealing probably removed the mechanical stresses generated during deposition, which leads to stabilization of the structure and reduction of lattice parameters, since the stresses in the coating can change the distance between atoms.

In general, a decrease in lattice parameters after annealing indicates that the heat treatment has stabilized the structure, reduced internal stresses, or changed its composition, which can improve the mechanical properties of the coating, such as hardness or wear resistance.

The study of the mechanical properties of such coatings by the most expressive and universal characteristic – hardness – shows that for thicknesses of 10 nm and more, there is a dependence that consists in increasing hardness with increasing layer thickness, when the contribution of interfacial interaction is minimized.

The average values (for ten measurements) of the hardness of MoN/CrN coatings measured on the device are: for samples obtained at $P_N = 3 \times 10^{-3}$ mm Hg, $U_b = -100$ V, HV$_{0.05}$ = 25.6 GPa, in the case of $U_b = -200$ V, HV$_{0.05}$ = 28.8 GPa. At annealing T = 700 °C for coatings obtained at $P_N = 3 \times 10^{-3}$ mm Hg, $U_b = -100$ V, HV$_{0.05}$ = 24.3 GPa, in the case of $U_b = -200$ V, HV$_{0.05}$ = 30.8 GPa.

At the same time, an increase in hardness is observed in the coatings obtained by continuous rotation, which can be attributed to both the size effect and the formation of an incoherent (with different types of crystal lattices) interfacial boundary between the layers [11].

4. CONCLUSIONS

1. Multilayer MoN/CrN coatings obtained by vacuum arc deposition obtained at P_N = 0.53 Pa, had a thickness of $\lambda \sim 145$ nm at $U_b = -100$ V, and at $U_b = -200$ V the thickness was $\lambda \sim 120$ nm.

2. The study of the nitrogen content in the coatings shows that the supply of the shear potential is reflected in the change in the N content in the coatings. This is associated with the selective sputtering of light nitrogen atoms from the coating surface. With an increase in the negative bias potential modulus U_b to – 200 V, an increase in the N content in the coatings is observed, while the amount of molybdenum and chromium decreases, which determines an increase in the efficiency of nitride formation.

3. The results of the study of the phase composition of the ion-plasma condensate MoN/CrN material at the vacuum annealing temperature at T = 700 °C remain practically unchanged and correspond to the initial post-condensation state.

4. The average hardness values of the MoN/CrN coatings are: for samples obtained at P_N = 0.53 Pa, U_b = – 100 V, $HV_{0.05}$ = 25.6 GPa, in the case of U_b = – 200 V, $HV_{0.05}$ = 28.8 GPa. When annealing at T = 700 °C for coatings obtained at P_N = 0.53 Pa, U_b = – 100 V, $HV_{0.05}$ = 24.3 GPa, in the case of U_b = – 200 V, $HV_{0.05}$ = 30.8 GPa.

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Вплив екрануючого потенціалу на процес формування наноструктурованих нітридних покриттів системи MoN/CrN

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Методом вакуумно-дугового осадження синтезовано багатошарові нітридні покриття MoN/CrN за двох різних потенціалів зміщення підкладки, а саме $U_{зс}$ = – 100 В та $U_{зс}$ = – 200 В. Покриття осаджували за робочого тиску азоту P_N = 0,53 Па. Систематично досліджено структурні та механічні властивості отриманих покриттів, особливу увагу приділено мікротвердості та термічній стабільності. Дослідження показало, що збільшення потенціалу зміщення від – 100 В до – 200 В призвело до значного підвищення твердості покриттів. Зокрема, покриття, осаджені при $U_{зс}$ = – 200 В, продемонстрували на 23,5 % вищу твердість порівняно з покриттями, отриманими при $U_{зс}$ = – 100 В, що вказує на покращене ущільнення і, можливо, більш тонкі мікроструктурні особливості, індуковані бомбардуванням іонами з вищою енергією. Виміряні значення мікротвердості (HV0,05) осаджених покриттів становили 25,6 ГПа для $U_{зс}$ = – 100 В і 28,8 ГПа для $U_{зс}$ = – 200 В. Дані значення підтверджують позитивний вплив більшого негативного зміщення підкладки на механічні характеристики багатошарової архітектури. Крім того, після осадження було проведено відпал при T = 700 °C для оцінки термічної стабільності та можливих фазових перетворень. Важливо, що відпал не призвів до збільшення твердості; навпаки, спостерігалося незначне зменшення середньої мікротвердості для покриття, осадженого при $U_{зс}$ = – 100 В, причому значення зменшилося до 24,3 ГПа. Однак для покриття, отриманого при $U_{зс}$ = – 200 В, відпал призвів до незначного збільшення твердості до 30,8 ГПа, що свідчить про підвищену термічну стабільність і стійкість до розм'якшення. Отримані результати підкреслюють важливість параметрів осадження для налаштування характеристик багатошарових нітридних покриттів для високотемпературних і зносостійких застосувань.

Ключові слова: Вакуумно-дугове осадження, Багатошарові покриття, Твердість, Адгезійна міцність.