

SECTION 3

PHYSICS OF RADIATION AND ION-PLASMA TECHNOLOGIES

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EFFECT OF NITROGEN PRESSURE ON THE STRUCTURAL AND MECHANICAL PROPERTIES OF V-Mo-N COATINGS DEPOSITED BY CATHODIC ARC EVAPORATION

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The coatings of the V-Mo-N system were deposited by the cathodic arc evaporation method from the unfiltered plasma of two V and Mo cathodes. Electron microscopy, energy dispersive analysis, X-ray diffractometry, nanoindentation, and adhesion testing have been used to study the effect of nitrogen pressure during deposition on surface morphology, composition, structure, and mechanical properties of coatings. It was found that at a nitrogen pressure of 1 Pa the V₂₉Mo₂₁N₅₀ coating formed with a predominantly cubic c-(V,Mo)N structure and an insignificant fraction of hexagonal h-(Mo,V)N. With an increase in the pressure of the reaction gas to 2...3 Pa, a gradual increase in the concentration of nitrogen up to 54 at.% and molybdenum up to 26 at.% is observed, which leads to a slight increase in the hexagonal phase concentration. All coatings are characterized by a fairly good surface quality, high hardness of 30...32 GPa and adhesion to the steel substrate. The c-(V,Mo)N coating has better resistance to crack propagation, correlates with a maximum $H^3/E^2 - 0.18$, and combines high hardness and ductility.

INTRODUCTION

The process of cathodic arc evaporation is widely used to modify the surface of materials, including reactive deposition of hard coatings based on transition metal nitrides on the surface of tools and machine parts. Transition metal nitrides are ceramics with exceptional properties, including high hardness, wear resistance, high temperature stability, and chemical inertness [1]. Every year, the requirements for products in many areas of application become more rigorous, so classical binary materials such as TiN, CrN, MoN, ZrN can no longer satisfy them [2, 3]. Transition metal nitrides have high hardness, they, but like most ceramics, are quite brittle, which can lead to coating failure due to the formation and propagation of cracks under high thermal and mechanical stresses. Thus, for many applications, high brittleness is not desirable, and coatings must simultaneously be relatively ductile [4].

The results of recent studies in the field of materials science of protective coatings indicate the possibility of obtaining new nanostructured materials with significantly improved functional properties due to the use of multicomponent systems [5–7]. Some theoretical studies on the electronic origin of plasticity have shown that the combination of Group IV and V transition metal nitrides, including TiN and VN, with Group VI metal nitrides, MoN and WN, improves the plasticity of the B1-NaCl pseudobinary structure while maintaining hardness by increasing the concentration valence electrons [8–10]. In particular, such promising systems include V_{1-x}Mo_xN_y. Recent experimental studies confirm the theoretical conclusions that films of the V-Mo-N system in a fairly wide range of element concentrations and synthesis

temperatures demonstrate increased plasticity compared to the binary systems VN, Mo-N and reference TiN [10–12]. In addition, the V-Mo-N system is quite attractive due to its special tribological properties. Vanadium and molybdenum are capable of forming lubricating oxide films (MoO₃, V₂O₅, elevated temperature) at which makes self-lubrication possible under dry friction conditions [13]. Compared with Mo-N and VN films, Mo-V-N films not only have higher hardness and better wear resistance, but also exhibit a low friction coefficient at room temperature and elevated temperature up to 700 °C [11–15]. It means that the development of processes for the deposition of the V-Mo-N system coatings by the vacuum-arc method, which is widely used in industry, is a rather promising task.

It is known, that cathodic arc deposition of coatings is a complex multifactorial process consisting of several stages: plasma generation, its transfer to the deposition surface, interaction of condensed particles with the gaseous medium and the deposition surface, formation of a coating on the substrate. At each stage, the pressure of the reaction gas has a significant effect [2–4]. The purpose of this work was to investigate the effect of nitrogen pressure during cathodic arc deposition of V-Mo-N coatings on their composition, structure, and mechanical properties.

MATERIALS AND EXPERIMENTAL TECHNIQUE

The coatings of the V-Mo-N system were deposited by cathodic arc evaporation from unfiltered plasma on a modernized Bulat-6 installation [16]. Two cathodes with

a diameter of 60 mm from commercially pure V (99.99%) and Mo (99.9%) were installed opposite each other in the vacuum chamber. During deposition, the arc current was 85 A for the vanadium cathode and 100 A for the molybdenum cathode. The coatings were deposited on polished 18Cr10NiTi stainless steel substrates with a size of 20×20 mm and a thickness of 2 mm with an average roughness R_a of about 0.02 μm . The samples were placed on a planetary rotating holder in the center of the vacuum chamber at a distance of 300 mm from each of the cathodes. During the deposition of coatings, the rotation speed of the substrate holder was 30 rpm.

The vacuum chamber was evacuated to a pressure of $2 \cdot 10^{-3}$ Pa. The samples were ion etched, using a constant bias potential on the substrate of -1300 V for 3 min. To improve film adhesion, a sublayer of V (about 0.1 μm thick) was deposited on the substrate for 5 min at a bias potential of -100 V. Nitrogen was used as a reactive gas during the deposition of V-Mo-N coatings, the pressure of which was 1, 2, or 3 Pa. The temperature of the substrate was maintained at about 500 °C. For all coatings deposition time was 60 min, and a substrate bias voltage -100 V. The thickness of the deposited coatings was about 10 μm .

The surface microstructure of the coatings and their elemental composition were studied using a JSM-7001F scanning electron microscope equipped with an attachment for X-ray energy-dispersive spectroscopy. The phase composition of the coatings was determined by X-ray diffraction analysis on a DRON-3 diffractometer in filtered Cu-K α radiation ($\lambda_{\text{Cu-K}\alpha} = 0.154178$ nm). The diffraction patterns were taken in the θ -2 θ scanning scheme in the range of diffraction angles of 20...120 degrees. The JCPDS database was used to interpret the obtained results. The size of crystallites in coatings (zones of coherent scattering) was calculated based on the expansion of diffraction lines using the Scherrer relation.

The hardness (H) and elastic modulus (E) of the coatings were determined by nanoindentation using a Nano Indenter G200 system (Agilent Technologies, USA) with a Berkovich diamond indenter. The indenter immersion depth was about 500 nm.

The adhesion of coatings to the substrate and the nature of their destruction during loading were assessed by studying electron microscopic images of indentations that appear on the surface of coatings when indenters are pressed at hardness testers. First, the method of the

Rockwell-C adhesion test using the TS-1 hardness tester was applied [17]. A spherical tip indenter was pressed at 1470 N load into a coating. Adhesion was determined by visual inspection of a microscopic image of the surface around the indentation. The adhesion rating scale has 6 grades: from HF1 (the highest level, no signs of peeling of the coating around the indentation. There may be a small number of shallow cracks outside the print) to HF6 (the lowest level – complete peeling of the coating to the substrate). Additional assessment of crack propagation in coatings during deformation was carried out using an LM 700 AT (Leco) microhardness tester with a Vickers indenter at a load of 10 N.

EXPERIMENTAL RESULTS

Electron microscopic images of the surface of V-Mo-N coatings, which were deposited at different nitrogen pressures in the chamber from 1 to 3 Pa, are shown in Fig. 1. It can be seen that the surface of all coatings is quite smooth, recreates the substrate surface relief and has a cellular structure with a small number of defects in the form of cathode material macroparticles, which is inherent in coatings deposited from unfiltered vacuum arc plasma. For all coatings, the diameter of macroparticles does not exceed 1 μm , which is a fairly good indicator. As the nitrogen pressure increases, the average size of macroparticles on the coating surface decreases, which is typical for deposition in the presence of active gasses in the vacuum chamber. This is usually associated with the formation of films of refractory compounds on the cathode surface [19].

It is known that with increasing pressure in the chamber, the interaction of plasma components with gas molecules increases, which ultimately affects the composition of the coatings. At low pressure, the interaction of plasma ions and electrons with gas is insignificant, and nitrogen enters the substrate mainly in the molecular state. With an increase in pressure, especially in the presence of a magnetic field, nitrogen activation (excitation, dissociation, and ionization of molecules) is possible, which enhances the processes of nitrogen binding in the coating [19]. The results of determining the elemental composition of the coatings are shown in Table.

In the $V_{29}Mo_{21}N_{50}$, which were deposited at a nitrogen pressure of 1 Pa, the nitrogen concentration is 50 at.%, that is, the ratio of metal atoms and nitrogen atoms is 1/1.

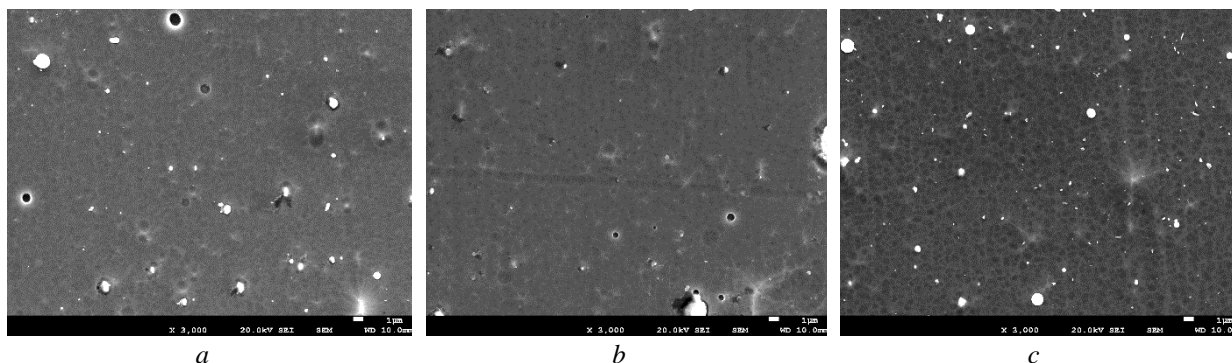


Fig. 1. SEM images of the V-Mo-N coatings surfaces, which were deposited at various nitrogen pressure in a vacuum chamber: a – 1; b – 2, and c – 3 Pa

An increase in the pressure of the reaction gas during deposition by a factor of three in the range from 1 to 3 Pa leads to the expected gradual increase in the nitrogen concentration in the coatings, but this increase is quite insignificant from 50 to 54 at.%. A stronger change in pressure has an effect on the content of metal components. In the sample deposited at a nitrogen pressure of 1 Pa, the concentration of vanadium is higher

than that of molybdenum – 29 and 21 at.%, respectively. With increasing pressure, the concentration of vanadium decreases, while that of molybdenum increases, which may be due to stronger scattering of lighter vanadium ions, which occurs on the plasma path from the cathode to the substrate. At a nitrogen pressure of 3 Pa, the concentration of vanadium is 20 at.%, and molybdenum – 26 at.%.

Elemental composition, structure and mechanical characteristics of V-Mo-N coatings depending on nitrogen pressure

Parameter	Nitrogen pressure, Pa		
	1	2	3
Composition, at.%	V ₂₉ Mo ₂₁ N ₅₀	V ₂₃ Mo ₂₅ N ₅₂	V ₂₀ Mo ₂₆ N ₅₄
Crystallite size, nm	9.5	8.0	7.7
Lattice parameter (of cubic phase), nm	0.4199	0.4204	0.4208
H, GPa	32±3	30±2	31±2
E, GPa	425±25	400±10	450±20
H/E	0.07	0.07	0.07
H ³ /E ² , GPa	0.18	0.17	0.15
Adhesion	HF1	HF1	HF2

According to the data of X-ray diffraction analysis, all obtained V-Mo-N coatings have a similar phase composition, despite differences in elemental composition. The X-ray diffraction patterns of the coatings obtained under different nitrogen pressure are almost identical (Fig. 2). The main phase in the coatings is a polycrystalline cubic solid solution c-(V,Mo)N, structural type NaCl (B1). The diffraction patterns show two broad peaks near 43.0 and 94.4 degrees, which correspond to the (200) and (400) reflections of the cubic solid solution. There are no other diffraction lines of this phase in the diffraction patterns, which is associated with the formation of a strong axial texture with the [100] axis in the direction normal to the sample surface. A similar texture was observed in V_{0.5}Mo_{0.5}N_x polycrystalline magnetron deposited coatings on MgO(001) substrates at temperatures ranging from 100 to 500 °C [11]. An increase in nitrogen pressure during coating deposition leads to a slight shift of the solid solution lines towards smaller angles and their expansion, which indicates an increase in the crystal lattice parameter and a decrease in the size of the coherent scattering region (crystallite size). The results of determining these parameters are presented in Table. With an increase in nitrogen pressure from 1 to 3 Pa, the crystal lattice parameter of the cubic solid solution c-(V,Mo)N gradually increases from 0.4199 to 0.4208, and the crystallite size decreases from 9, 5 to 7.7 nm.

The possibility of formation of cubic solid solutions in the V-Mo-N system with a wide range of element concentrations is largely due to the fact that the VN (JCPDS No. 35-0768) and γ -Mo₂N (JCPDS No. 25-1366) compounds have a similar crystal structure. The metal atoms are located at the sites of the fcc lattice, while the nitrogen atoms occupy the octahedral voids of the base metal, and in the case of molybdenum, only half of the voids are filled [20]. The formation of solid solutions with a cubic structure of the NaCl (B1) type was observed in a number of works [10–12, 14], in particular, in V_{0.5}Mo_{0.5}N_x, which were deposited by reactive

magnetron sputtering on Al₂O₃ (0001). The structure was retained despite a significant decrease in the nitrogen content (x) in the films from x = 0.9 at a substrate temperature of 100 °C to x = 0.4 at a temperature of 900 °C [11].

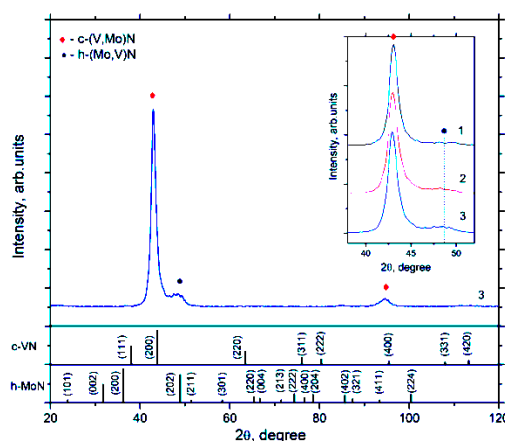


Fig. 2. X-ray diffraction patterns of samples with V-Mo-N coatings. On the tab, sections of diffraction patterns for coatings obtained at different nitrogen pressures: 1 – 1; 2 – 2, and 3 – 3 Pa

As can be seen from Fig. 2, along with the intense line (200) of the cubic solid solution, another weak and broad line is detected in the diffraction patterns of the coatings near 48°. On the diffraction pattern of the sample deposited at a nitrogen pressure of 1 Pa, it is almost invisible. With an increase in the nitrogen pressure during deposition, which leads to an increase in the Mo content in the coatings, this line becomes more distinct. Most likely, this line belongs to the h-(Mo,V)N phase, a solid solution based on hexagonal δ -MoN (JCPDS No. 25-1367). According to the data of [13], such a hexagonal phase is the main one in coatings with a high content of molybdenum, when the vanadium concentration does not exceed 26 at.%. In this case, the main phase is the cubic c-(V,Mo)N, and the hexagonal h-(Mo,V)N is a small volume fraction.

The mechanical characteristics of the coatings determined by nanoindentation are shown in Table. The table shows the values of hardness H , Young's modulus E , as well as the calculated values H/E and H^3/E^2 for coatings obtained at various nitrogen pressures. It can be seen that despite different nitrogen pressure during deposition the coatings show similar mechanical characteristics with a high hardness of 30...32 GPa and a Young's modulus in the range of 400...450 GPa. The obtained hardness values are significantly higher than for coatings of the V-Mo-N system, which were obtained by magnetron sputtering. For magnetron coatings, dependent on the component concentration, the hardness varies within 15...26 GPa [11–17].

The features of the structure and mechanical properties of the obtained coatings can be explained on the basis of the data presented in Ref. [14], which is devoted to the study of the nanostructural evolution of single-phase polycrystalline (B1) $V_{1-x}Mo_xN$ ($x \approx 0,4$). Films deposited by magnetron sputtering were subjected to annealing at temperatures from 700 to 1050 °C. Using the methods of dispersive X-ray spectroscopy, X-ray diffraction (XRD), transmission electron microscopy and molecular dynamics, the authors found that at an annealing temperature of 730 °C, the effect of film aging is observed due to the spinodal decomposition of a cubic solid solution into coherent strained domains rich in VN/MoN, which accompanied by an increase in hardness from 23 to 31 GPa. In films annealed at temperatures above 850 °C, decomposition leads to the formation of a hexagonal Mo_xN_y and cubic VN phase, which causes a decrease in hardness to 22 GPa.

In these experiments, a V-Mo-N coating with the c-(V,Mo)N solid solution structure was obtained, in which the components are at the limit of solubility. The high hardness of the coatings indirectly indicates a nanostructural state caused by spinodal decay similar to that occurring during annealing of magnetron films. We observed signs of such a phase transformation during deposition earlier for vacuum-arc coatings of the Ti-Al-N and Ti-Al-Si-N systems [21, 22]. The possibility of spinodal-type decay during the deposition of vacuum-arc coatings is due to the high energy of the vacuum-arc plasma ions forming the coating. It is known that when interacting with a solid body, ions with an energy of the order of hundreds of electron-volt characteristic of vacuum-arc deposition, nanometer thermal peaks arise – overheating regions near the ion trajectory in a substance, the temperature in which can reach several thousand degrees. In such thermal peaks, a significant acceleration of various kinetic processes is possible, aimed at minimizing the free energy of the system – structural transitions, diffusion of impurities, chemical reactions, relaxation of mechanical stresses, etc. [23, 24]. With an increase in the Mo and N concentration, which is observed with an increase in gas pressure during the deposition of V-Mo-N coatings, an increasing amount of these elements is not able to dissolve in the cubic lattice of a supersaturated c-(V,Mo)N solid solution and is isolated as a separate hexagonal h-(Mo,V)N phases. An increase in the amount of the hexagonal phase in the coatings will lead to a decrease in their hardness. In the considered nitrogen pressure range of 1...3 Pa, the

volume fraction of the hexagonal phase in the coatings remains insignificant, so the resulting coatings have similar mechanical characteristics.

It was noted above that high-quality wear-resistant coatings require a combination of hardness, ductility, and recovery ability. Hardness and elasticity are of equal importance for improving wear resistance, especially in complex processes associated with impact loading and erosion. An important feature of nitride coatings is that materials with high H can differ significantly in the values of the elastic modulus E . The combination of high hardness and low elastic modulus is considered optimal. The H/E ratio is often used to characterize the resistance of a material to elastic fracture [25], while the H^3/E^2 can characterize the ability to resist initiation cracks and cracking [26]. It can be seen from Table that the H/E values for all obtained coatings do not differ within the error and are equal to 0.07, which is typical for nanostructured materials and indicates a fairly high ability of the coating to resist damage and elastic deformation. For most V-Mo-N coatings obtained by magnetron sputtering, the H/E ratio has a smaller value within 0.05...0.06 [11–13]. As for the ratio H^3/E^2 , it can be seen that with an increase in nitrogen pressure during deposition, this indicator decreases from 0.18 to 0.15. Thus, the maximum value of the parameter H^3/E^2 , i.e. the expected resistance to crack propagation is found for the hardest coating deposited at a nitrogen pressure of 1 Pa, having the smallest volume hexagonal phase fraction.

Indentation tests have been used to qualitatively assess the adhesion of the coatings to steel substrates and evaluate the propagation of cracks in coatings under the load. The imprints that appeared on the surface of the coatings after immersion of the indenters were observed using scanning electron microscopy. In Fig. 3 are shown the images of indentations in Rockwell test (see Fig. 3,a,c,e) and Vickers test (see Fig. 3,b,d,f) for V-Mo-N coatings, deposited at different nitrogen pressures.

Evaluation using Rockwell-C tests (see Fig. 3,a,c,e) shows that all coatings have high adhesion to the steel substrate and deform with it under load. There are no chips outside the indentations and only slight radial cracks around the edge of the indentation are found, which indicates a good plasticity of the coatings. Inside the indentations, both radial and circular cracks are observed, the length and depth of which increase with increasing nitrogen pressure during coating deposition. Based on the number and size of cracks for coatings deposited at a pressure of 1 and 2 Pa, the level of their adhesion can be assessed by class HF1, and at a pressure of 3 Pa – HF2 [18].

On the surface of the coatings after Vickers microindentation, there are also no chips or delaminations. The coating deforms together with the substrate, and thin cracks appear on their surface (see Fig. 3,b,d,f). The mechanisms of induced cracking during quadrangular pyramid indentation are generally classified into four categories described by Richter [27] radial cracks directed along the diagonals of the indentation (type I), external circumferential cracks (type II), internal circumferential cracks (type III), and radial “claw” cracks (type IV).

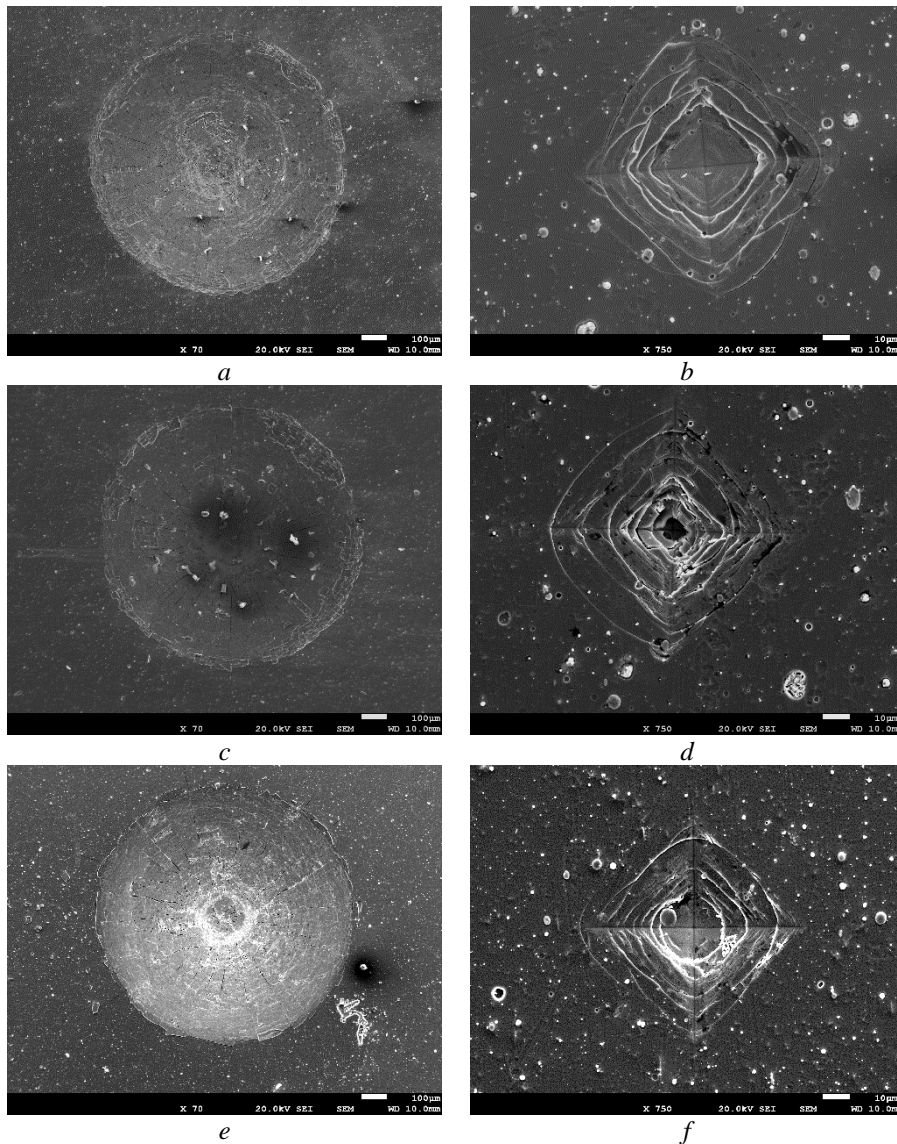


Fig. 3. SEM images of indentations after the Rockwell (a, c, e) and Vickers (b, d, f) tests on the V-Mo-N coatings surfaces deposited on steel substrates at various nitrogen pressures: a, b – 1; c, d – 2, and e, f – 3 Pa

For all V-Mo-N coatings, only internal circumferential cracks of type III are observed repeating the shape of the imprint. Such cracks are characteristic for ceramic coatings, in particular TiN, and the increase in their number is explained, first of all, by the deterioration of ductility. It can be seen that less other cracked coatings are deposited at a minimum nitrogen pressure of 1 Pa. With increasing pressure during deposition, the density of cracks within the imprint increases. Thus, all obtained V-Mo-N coatings with a predominantly cubic c-(V,Mo)N structure and a small fraction of hexagonal h-(Mo,V)N demonstrate a combination of high hardness and plasticity. Somewhat better resistance to crack propagation has a coating of $V_{29}Mo_{21}N_{50}$ deposited at a nitrogen pressure of 1 Pa, in which the content of the hexagonal phase is the lowest, correlating with the maximum value of the parameter $H^3/E^2 = 0.18$.

CONCLUSIONS

Using the methods of electron microscopy, energy dispersive analysis, X-ray diffraction, nanoindentation, and adhesion tests, the influence of nitrogen pressure

changes during deposition in the range 1...3 Pa on the surface morphology, composition, structure, and mechanical properties of V-Mo-N coatings was investigated.

It was found that at a nitrogen pressure of 1 Pa, a $V_{29}Mo_{21}N_{50}$ with a predominantly cubic structure of the c-(V,Mo)N solid solution (structural type B1) and an insignificant hexagonal fraction of h-(Mo,V)N. With an increase in the pressure of the reaction gas, a gradual increase in the concentration of nitrogen to 54 at.% and molybdenum to 26 at.% is observed, which leads to a slight increase in the hexagonal phase concentration. All coatings have a fairly smooth surface with a small number of defects in the form of macroparticles up to 1 µm in diameter.

Using the nanoindentation method, it was determined that despite different nitrogen pressure during deposition all investigated are coatings are characterized by similar mechanical properties with a high hardness of 30...32 GPa and a Young's modulus in the range of 400...450 GPa. The obtained values are higher than for coatings of the V-Mo-N system obtained by magnetron sputtering.

The results of the study of electron microscopic images of indentations that appear on the surface of the coatings after indentation according to Rockwell and Vickers tests indicate a high adhesion of the coatings to the steel substrate. It is shown that c-(V,Mo)N coatings provide a combination of high hardness and ductility under load. The coating with the minimum hexagonal phase concentration has the best resistance to crack propagation, correlates with the maximum value of the parameter $H^3/E^2 - 0.18$.

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ВПЛИВ ТИСКУ АЗОТУ НА СТРУКТУРУ ТА МЕХАНІЧНІ ВЛАСТИВОСТІ ПОКРИТТІВ V-Mo-N, СИНТЕЗОВАНИХ МЕТОДОМ ВАКУУМНО-ДУГОВОГО ОСАДЖЕННЯ

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Покриття системи V-Mo-N осаджували вакуумно-дуговим методом з нефільтрованої плазми двох катодів з V та Mo. Методами електронної мікроскопії, енергодисперсійного аналізу, рентгенівської дифрактометрії, наноіндентування та адгезійного тестування досліджено вплив зміни тиску азоту при осадженні на морфологію поверхні, склад, структуру та механічні властивості покриттів. З'ясовано, що при тиску азоту 1 Па формується покриття $V_{29}Mo_{21}N_{50}$ з переважно кубічною структурою c-(V,Mo)N і незначною часткою гексагонального h-(Mo,V)N. Збільшення тиску реакційного газу до 2...3 Па викликає поступове зростання концентрації азоту до 54 ат.% і молібдену до 26 ат.%, що призводить до невеликого збільшення вмісту гексагональної фази. Усі покриття характеризуються достатньо гарною якістю поверхні, високими показниками твердості (30...32 ГПа) та адгезії до сталеві підкладки. Покриття c-(V,Mo)N демонструють при навантаженні кращу стійкість до поширення тріщин, що корелює з максимальним значенням параметра H^3/E^2 – 0,18, і також поєднання високої твердості та пластичності.