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Features of the Formation of the Structure and Phase Composition of Modified Chrome PVD Coatings

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The results of studies of the structure and phase composition of thick chromium coatings on the surface of the steel obtained by vacuum-arc deposition (arc-PVD) in an arc discharge and modified with nitrogen are presented. The modification is carried out directly in the process of vacuum-arc deposition in an argon-gas environment containing nitrogen. The features of the formation of the structure and phase composition of chromium coatings depending on the percentage of nitrogen in the chamber (from 20 to 100%) and the effect of the pulsed deposition mode at a fixed pressure value within the chamber ($(3-7) \cdot 10^{-2}$ Pa) and substrate temperature (520–550°C) are established. The analysis of the studies shows that chromium coatings obtained in a steady-state mode in an argon environment (without nitrogen) have an open texture: in the cross section, the structure is columnar and coarse-crystalline, has an explicit relief surface with the release of fairly large pyramidal crystallites and a small droplet phase. The use of a pulsed deposition mode in an argon environment weakens significantly the texture of the coating, ‘smooths’ the surface with the formation of a fine-crystalline, close to globular coating structure. The introduction of nitrogen into the argon-gas environment changes the structure and phase composition of the coatings. With an increase

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in the percentage of nitrogen within the chamber, the structure of the coating in the cross-section changes from columnar coarse-crystalline to globular fine-crystalline, the lattice parameter and the size of the coherent-scattering region decrease, and the dislocation density increases. The introduction of nitrogen into the argon-gas environment leads to the formation of the hexagonal close-packed phase Cr_2N and the face-centred cubic phase CrN , the amounts of which depend on the deposition parameters. The use of a pulsed deposition mode, when modifying the coating with nitrogen, allows increasing the amount of nitrides in the coating compared to the steady-state mode.

Key words: arc-PVD, chromium coating, structure, phase composition, nitriding.

Представлено результати досліджень структури та фазового складу товстих хромових покриттів на поверхні криці, одержаних методом вакуумно-дугового осадження (arc-PVD) у дуговому розряді та модифікованих Нітрогеном. Модифікування проводилося безпосередньо в процесі вакуумно-дугового осадження в середовищі аргону, що містить азот. Встановлено особливості формування структури та фазового складу хромових покриттів залежно від відсотка Нітрогену у камері (від 20 до 100%) та впливу імпульсного режиму осадження за фіксованих значень тиску у камері $((3-7) \cdot 10^{-2} \text{ Па})$ та температури підкладки (520–550°C). Аналіза досліджень показала, що хромові покриття, одержані за стаціонарного режиму в середовищі аргону (без азоту), мають яскраво виражену текстуру: у поперечному перерізі структура є стовпчастою, крупнокристалічною, має виражену рельєфну поверхню з виділенням достатньо великих пірамідальних кристалітів і дрібної крапельної фази. Використання імпульсного режиму осадження в середовищі аргону значно послаблює текстуру покриття, «згладжує» поверхню з утворенням дрібнокристалічної, близької до глобулярної структури покриття. Введення азоту в середовище аргону змінює структуру та фазовий склад покриттів. Зі збільшенням відсотка Нітрогену у камері структура покриття в поперечному перерізі змінюється від стовпчастої крупнокристалічної до глобулярної дрібнокристалічної, зменшуються параметер кристалічних ґратниць і розмір області когерентного розсіювання, а густина дислокацій збільшується. Введення азоту в середовище аргону приводить до утворення гексагональної щільноупакованої фази Cr_2N і гранецентрованої кубічної фази CrN , кількість яких залежить від параметрів осадження. Використання імпульсного режиму осадження під час модифікування покриття Нітрогеном уможливило збільшити кількість нітридів у покритті порівняно зі стаціонарним режимом.

Ключові слова: електродугове нанесення покриття фізичним осадженням парів, хромове покриття, структура, фазовий склад, азотування.

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1. INTRODUCTION

Thick chromium-based coatings are widely used as protective coatings:

as corrosion-resistant, antifriction and wear-resistant coatings [1–6]. The galvanic method of applying thick chromium coatings has long been one of the most widely used. The advantages of this method include its technical simplicity and the ability to obtain fairly thick and hard coatings [7–8]; the disadvantages include poor adhesion and porosity of the coating. Galvanic production is one of the most dangerous in terms of emissions of harmful substances into the environment [9], therefore, interest in environmental friendly technologies for obtaining coatings, in particular PVD technologies, has recently increased. One of the types of PVD technologies that allow obtaining the sufficiently thick chromium-based coatings (more than 10 μm thick) is the deposition of coatings by condensation from the gas phase in a vacuum arc discharge (arc-PVD) [10–12]. The properties of coatings primarily depend on their structure and phase composition. Arc-PVD allows controlling the structure of the coating with technological process parameters and achieving high adhesion to the substrate [13–15]. Having a number of advantages over galvanic coatings, PVD chrome coatings are still inferior to them in hardness. One of the ways to improve the mechanical characteristics of chrome PVD coatings is to modify them with nitrogen. Chromium nitrides can be classified as one of the most popular materials used in surface engineering. This is primarily due to the fact that they are characterized by very good tribological and mechanical properties [16, 17]. Compared to titanium nitrides, chromium nitride coatings have lower thermal conductivity, greater resistance to oxidation at high temperatures and greater plasticity, and have good corrosion and chemical resistance [18, 19]. Studies [20–25] have shown that the properties of Cr–N coatings are fundamentally determined by their structure and phase composition. Thus, expanding knowledge about the patterns of formation of the structure and phase composition of chromium coatings depending on the parameters of vacuum-arc deposition is relevant.

2. MATERIALS AND METHODS

Vacuum-arc deposition was carried out in a specialized vacuum plant with an end-face electric arc evaporator of metals. The cathode was made of chromium in the form of a cylinder with a diameter of 50 mm. The coatings were deposited on flat samples made of 38XH3MΦA (analogue 34NiCrMoV14-5) steel. Before applying the coating, chemical and mechanical cleaning of the samples was carried out. The samples were located in a plane parallel to the end surface of the cathode at a distance of 120 mm. The air was pumped out of the chamber until a deep vacuum $P = 1.3 \cdot 10^{-2} - 4 \cdot 10^{-3}$ Pa was achieved. After pumping out, the working gas (argon, argon + nitrogen or nitrogen depending on the technological process) was introduced into the vacuum chamber to a

pressure of $P = (5.3\text{--}6.7) \cdot 10^{-2}$ Pa. The steady-state deposition mode was carried out at a constant arc burning current of 70 A, arc burning voltage of 30–40 V, without bias voltage on the cathode, and the substrate temperature of 520–550°C. The pulsed deposition mode consisted of alternating cycles of low bias voltage of 40 V on the substrate lasting 5 minutes with a short pulse of 400 V lasting 20 s. The discharge current, arc burning voltage, chamber pressure, and process temperature were maintained the same as in the steady-state deposition mode.

Metallographic studies of the surface and cross-section were carried out using a metallographic light microscope MIM-10 (magnification up to $\times 1200$). The structure of the coating was revealed by electrolytic etching of the ground surface and cross-section of the coatings on an electrolytic polishing machine 'Electro-P' (DEVCO S.r.l) in a 10% solution of oxalic acid.

X-ray diffraction analysis (XRD) was performed on a DRON-2 X-ray diffractometer in monochromatic CoK_α radiation with a wavelength of $\lambda = 1.7902$ Å. Chemical elements and phases were identified by comparing interplanar distances with the PCPDFWIN card index. The parameters of the crystal lattice, microstrains, and defect density were calculated using the Williamson–Hall method. The sizes of coherent scattering regions (CSRs) were determined using the Selyakov–Scherrer formula. The textured state was assessed by comparing the relative intensities of diffraction peaks with reference values.

3. RESEARCH RESULTS AND DISCUSSION

The influence of the percentage ratio of nitrogen and argon in the gas environment of the vacuum chamber, as well as the use of a pulsed deposition mode of the change in the structure and phase composition of vacuum-arc chromium coatings was studied. The surface of the chromium coating obtained in a steady-state deposition mode in a pure argon environment is relief with the release of fairly large pyramidal crystallites with transverse dimensions of 5–7 μm and droplet inclusions with a diameter of 2–3 μm (Fig. 1, *a*).

Figure 1, *b* shows the surface structure after electrolytic etching, *i.e.*, crystallites with a certain degree of faceting emerges on the surface, which indicate textured growth. The structure in the cross section is columnar with small inclusions of equiaxed crystals, which disrupt the characteristic normal growth of the coating (Fig. 1, *c*).

To describe the process of formation of the structure of ion-plasma coatings, various known models of structural zones (MSZ) are used [26, 27]. The variety of such models is great and each of the models takes into account different factors that influence the mechanism of the initial stages of coating formation and its evolution. The main such factors include: homologous temperature T_s/T_m (the ratio of the sub-

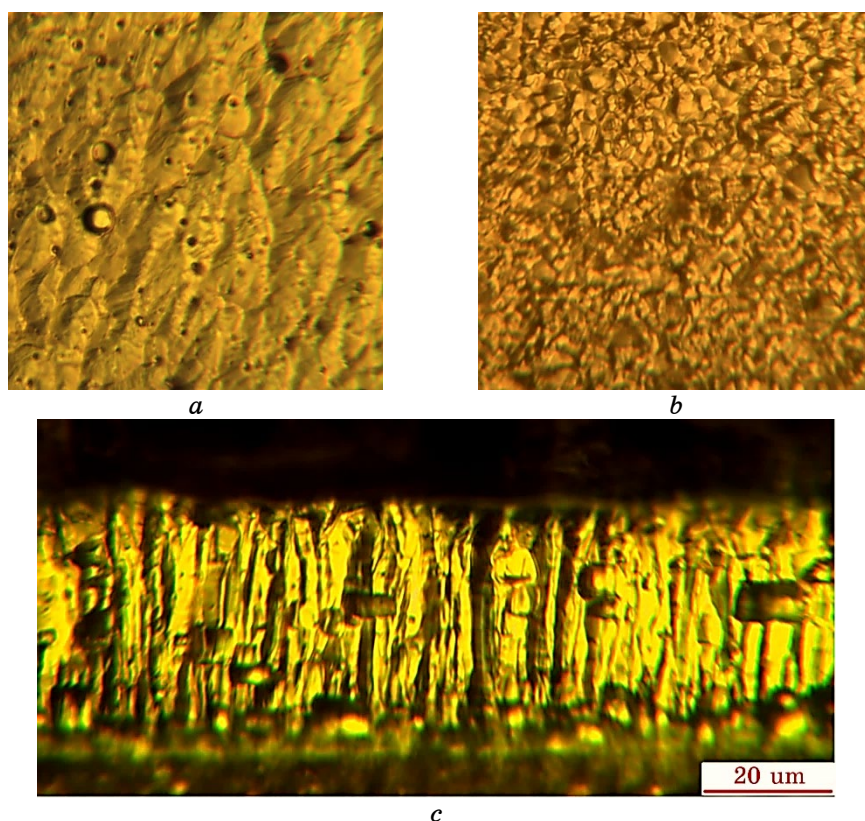


Fig. 1. Surface morphology (*a*), surface structure (*b*), cross-sectional structure (*c*) of the base chromium coating obtained in a steady-state deposition mode in a pure argon environment.

strate temperature to the melting point of the deposited coating material in kelvins), chamber pressure, arc burning current, substrate bias voltage, distance between the cathode and the substrate and their geometry, and impurity concentration in the chamber gas environment.

In this work, we investigated the effect of nitrogen concentration in the chamber on the coating formation process, so we chose the Barna and Adamik MSZ [28] (Fig. 2) as a basis, according to which an important role in the formation of the coating structure is played with the homologous temperature and the presence of active impurities that are released during the structuring and restructuring of the coatings.

In this study, the homologous deposition temperature fluctuated in a narrow range of $T_s/T_m \cong 0.36\text{--}0.38$, *i.e.*, was practically constant. The cross-sectional structure of the chromium coating obtained in the steady-state deposition mode in an argon environment (Fig. 1, *c*) correlates quite well with the structure similar to zone 3 (Fig. 2, *a*). At the

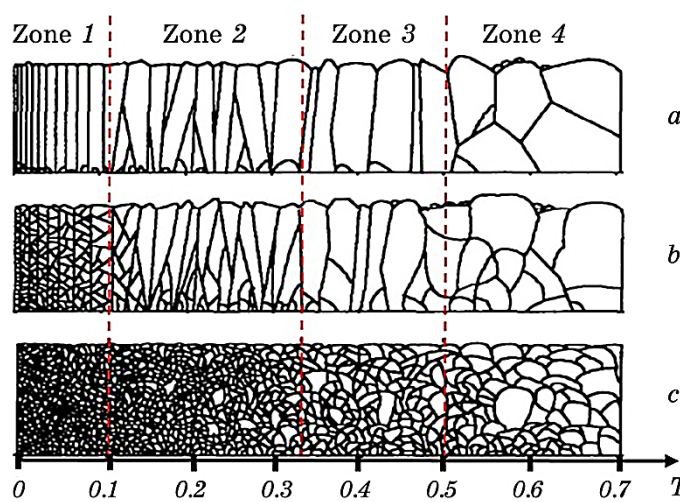


Fig. 2. Barna and Adamik MSZ: coating without impurity (base) (a), coating with impurity percentage of $\approx 1\%$ (b), coating with impurity percentage of $\geq 10\%$ (c).

substrate-coating interface, a transition layer is observed (Fig. 1, c), consisting of small misoriented crystallites.

The structure of the transition layer can be explained by the Stran-ski–Krastanov mechanism [29]. According to this mechanism, point nucleation centres are formed on the substrate, the rate of formation of which depends on the local temperature on the substrate, which then turn into two-dimensional islands, the growth rate of which depends on the surface diffusion of adatoms over the surface of the substrate. The relative rates of nucleation and formation of islands regulate the grain size before coalescence. At the initial stages of deposition, before coalescence, tangential growth predominates with the formation of equiaxed grains, and after coalescence, normal growth becomes pre-dominant.

The introduction of nitrogen into the chamber gas environment as an active impurity leads to a change in both the surface morphology and the cross-sectional structure of the chromium coatings. Figure 3 shows photographs of the morphology and surface structure of the chromium coating obtained in the steady-state deposition mode at the minimum (20%N₂80%Ar) and maximum (80%N₂20%Ar) percentage of nitrogen in the chamber gas environment.

As can be seen in Fig. 3, a, the surface of the coating deposited in a 20%N₂80%Ar gas mixture remains relief.

The crystallites emerging on the surface are smoothed and reduced to several microns. A subsequent increase in the nitrogen concentra-tion in the chamber to 80%N₂20%Ar ‘smooths’ the coating surface

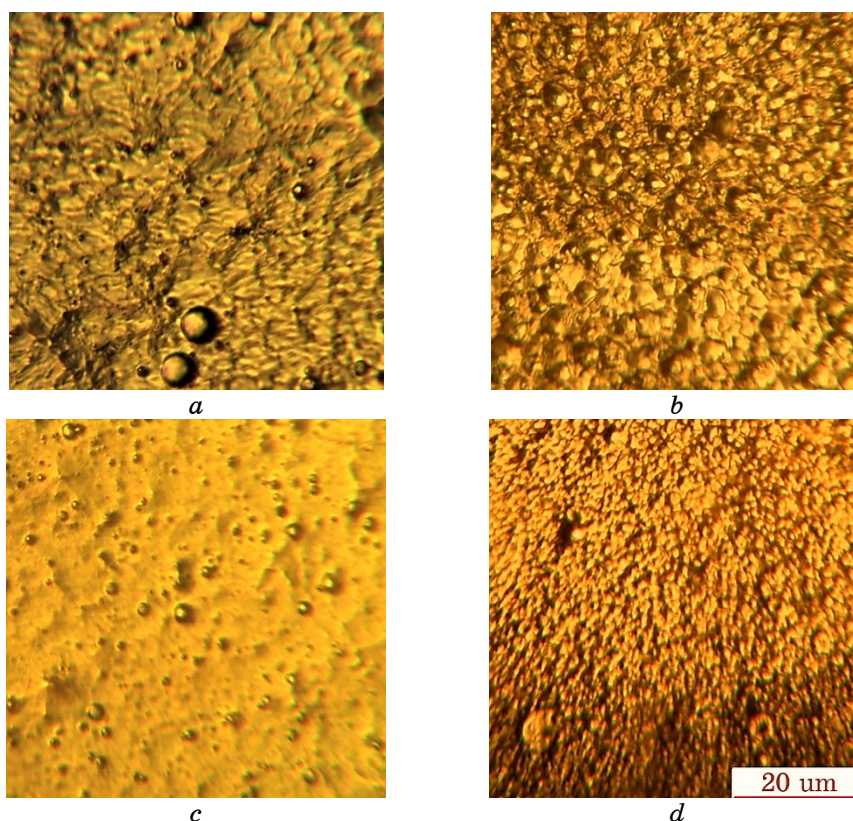


Fig. 3. Morphology and surface structure of chromium coatings obtained in a steady-state deposition mode in a nitrogen and argon environment with different percentage contents: 20%N₂80%Ar (*a, b*); 80%N₂20%Ar (*c, d*).

even more (Fig. 3, *c*). After electrolytic etching, globular elements are observed on the coating surface, the sizes of which decrease significantly with an increase in the nitrogen concentration in the chamber (Fig. 3, *b, d*).

According to the Barna and Adamik MSZ, the addition of impurities changes the coating structure. At low impurity concentrations ($\sim 1\%$), it accumulates predominantly at the grain boundaries during coating formation, resulting in a bimodal grain size distribution of the developing columnar structure; the diameter of the developing columns in zone 3 becomes smaller (Fig. 2, *b*) than in the base coating (Fig. 2, *a*). The texture, with minimized surface energy and interfacial energy, is not as explicit as in the base coating. With increasing impurity concentration, impurity segregation due to grain boundary motion becomes effective, resulting in complete coverage of the forming crystallite surface with a passivating layer and the development of three-

dimensional grains (Fig. 2, *c*), which are separated by stabilized grain boundaries. The impurity-stabilized grain size distribution is bimodal.

Similar conditions, in our opinion, are realized in this study during the deposition of chromium in the reaction gas nitrogen environment. Nitrogen ions, combining with chromium ions in the plasma volume, form nitrides, which act as impurities passivating the normal growth of crystallites, as evidenced by the structure of the coating in the cross section in Fig. 4.

The coating deposited from the 20%N₂80%Ar gas mixture is still characterized by a columnar structure interrupted by equiaxed grains (Fig. 4, *a*), but the diameter of the columns is significantly smaller than for the base coating (Fig. 1, *c*). With an increase in the nitrogen concentration in the chamber to 40%N₂60%Ar, the effect of suppressing columnar growth increases, globular grains with a disordered ori-

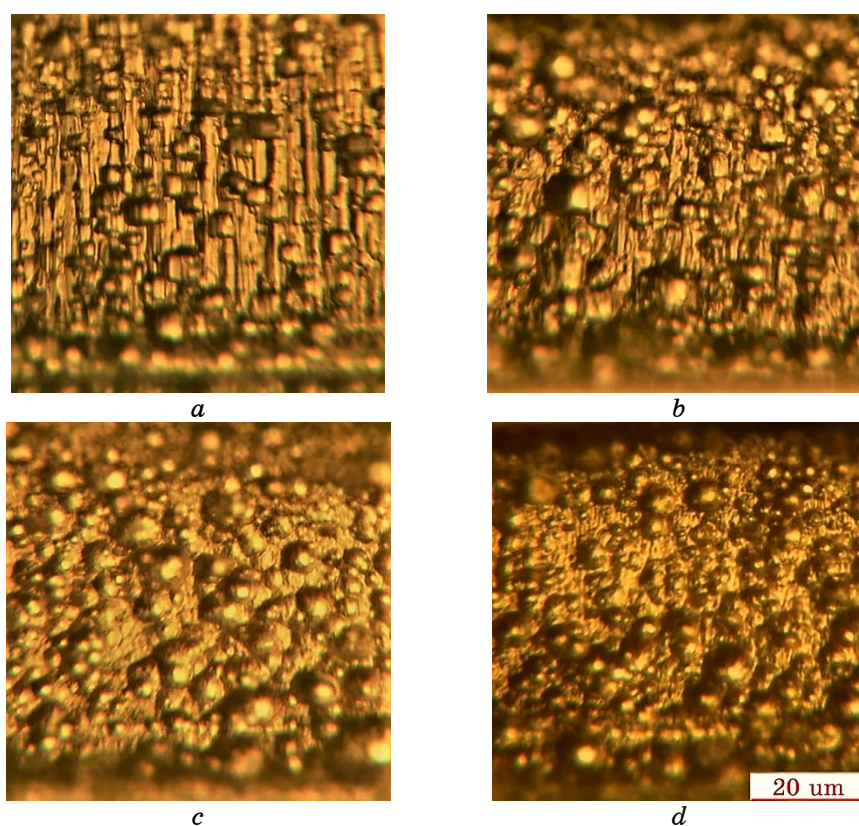


Fig. 4. The cross-sectional structure of chromium coatings obtained in a steady-state deposition mode in a nitrogen and argon environment with different percentage contents: 20%N₂80%Ar (*a*), 40%N₂60%Ar (*b*), 60%N₂20%Ar (*c*), 80%N₂20%Ar (*d*).

entation are formed (Fig. 4, *b*). A subsequent increase in the nitrogen concentration in the chamber gas mixture in the range of 60–80%N₂ completely disrupts the columnar growth of the coating and creates conditions for the formation of a globular structure and a decrease in the crystallite sizes (Fig. 4, *c, d*).

The study of coatings obtained in the steady-state deposition mode at 100%N₂ in the chamber showed a very low quality of such coatings. The coatings are friable, porous, and, during the study, they delaminate in planes parallel to the substrate, so they were of no interest for their further study. In the second stage of the study on the effect of deposition parameters on the structure and phase composition of chromium coatings, it was found that the use of pulsed deposition modes also allows one to 'escape' from the classical columnar structure of chromium condensates.

Figure 5 shows photographs of the surface morphology and structure of chromium coatings deposited in a pure argon environment in a pulsed mode.

Comparing the surface morphology of the chromium coating obtained in an argon environment in a steady-state mode (Fig. 1, *a*) and in a pulsed mode (Fig. 5, *a*), it is evident that the pulsed mode significantly smooths and refines the surface structure. After electrolytic etching, the release of small faceted crystallites is observed on the surface of the coating (Fig. 5, *b*). The cross-sectional structure (Fig. 5, *c*) is similar to the structure obtained in a steady-state mode with the addition of 40–60% nitrogen to the chamber, and consists of thin columns interrupted by globular grains.

Thus, the pulse mode plays the role of a passivating factor, similar to the effect of impurities during deposition in a nitrogen environment, which disrupts the columnar textured growth of the chromium coating.

Adding nitrogen to the chamber during the pulsed deposition mode leads to an effect similar to that for the steady-state mode—the surface morphology becomes smoothed cellular (Fig. 6, *a*), the surface structure is globular fine-grained (Fig. 6, *b*), in the cross section the structure is globular (Fig. 6, *c*). Increasing the nitrogen content to 80% leads to the formation of a layered structure (Fig. 6, *d*). The coating is very fragile, breaks down along the layer boundaries.

The results of XRD of chromium obtained in steady-state and pulsed deposition modes in an argon environment showed the presence of the body-centred cubic phase of Cr. Figure 7 shows the diffraction patterns of chromium coatings obtained in steady-state and pulsed deposition modes.

Analysis of the obtained data indicates the presence of a pronounced [200] texture in the chromium coating obtained in the steady-state mode.

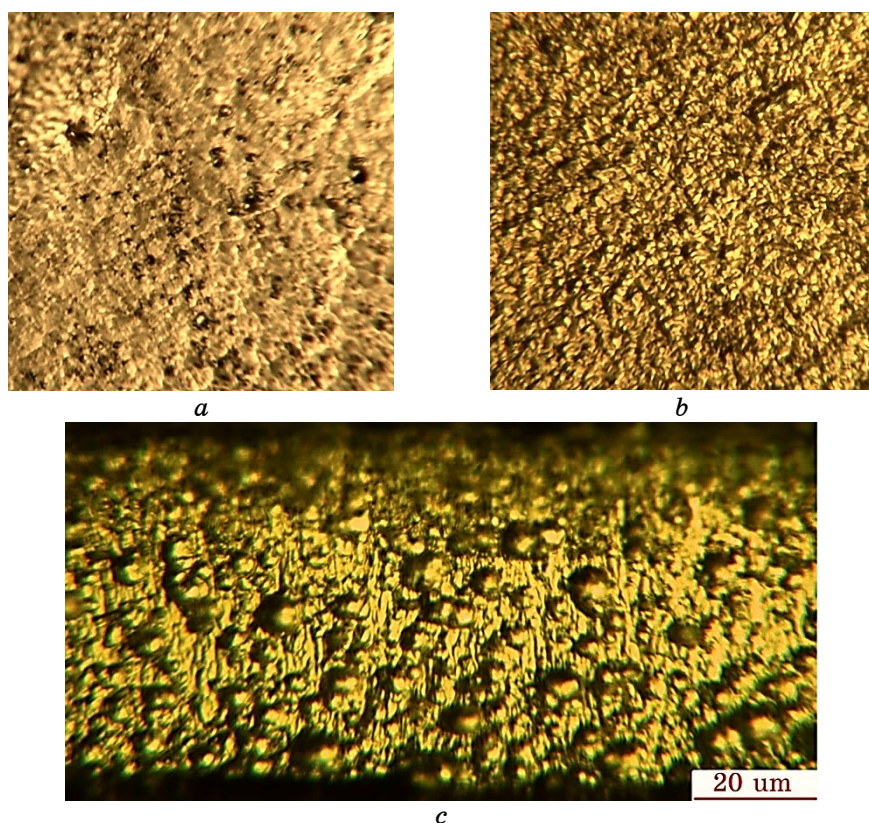


Fig. 5. Surface morphology (*a*), surface structure (*b*), cross-sectional structure (*c*) of a chromium coating obtained in a pulsed deposition mode in a pure argon environment.

The use of the pulsed mode leads to a redistribution of the intensities of the diffraction maxima, which can be used to estimate the weak $[200] + [211]$ texture.

The lattice parameter as a result of the pulse mode decreases from 2.8985 to 2.8879 Å, which indicates the occurrence of compressing stresses. A finer crystalline and more defective structure is formed with a CSRs size of 1340 nm and a dislocation density of $9.51 \cdot 10^{10} \text{ cm}^{-2}$ (for the steady-state mode, these parameters were equal to 1600 nm and $4.04 \cdot 10^{10} \text{ cm}^{-2}$, respectively). Thus, the use of the pulsed deposition mode leads to the suppression of textured growth and refinement of the chromium coating structure.

Adding nitrogen to the gas environment of the chamber led to a change in the microstructure, texture and phase composition of the coating of chromium coatings. Figure 8 shows the diffraction patterns

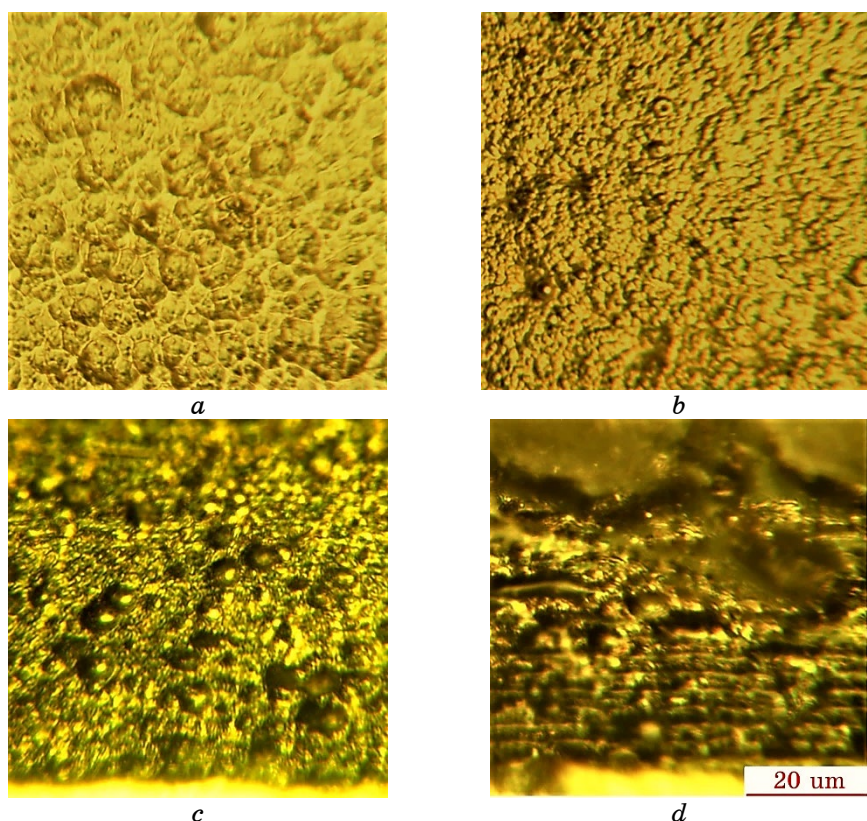


Fig. 6. Morphology (*a*), surface structure (*b*), cross-sectional structure (*c*) of a chromium coating obtained in the pulsed deposition mode in a gas environment of 60%N₂20%Ar and in a gas environment of 80%N₂20%Ar (*d*).

of chromium coatings modified with nitrogen. The texture of chromium modified with nitrogen in a steady-state mode at low nitrogen concentrations in the gas environment remains the same as for pure chromium: the most intense line remains (200). With increasing nitrogen concentration in the chamber, a redistribution of diffraction maximum occurs: the intensity of the maximum (200) decreases, while the maximum (110) and (211) are enhanced, which is associated with passivation of the surface of growing crystals by chromium nitrides. Phase XRD showed that modification of chromium with nitrogen leads to the formation of two chromium nitrides—Cr₂N with an hcp lattice and CrN with an f.c.c. lattice.

When modifying chromium with nitrogen in a steady-state mode in a chamber gas environment with a nitrogen percentage of less than 40%, chromium nitrides are practically not formed (< 1%). Increasing the nitrogen percentage in the chamber to more than 60% leads to an

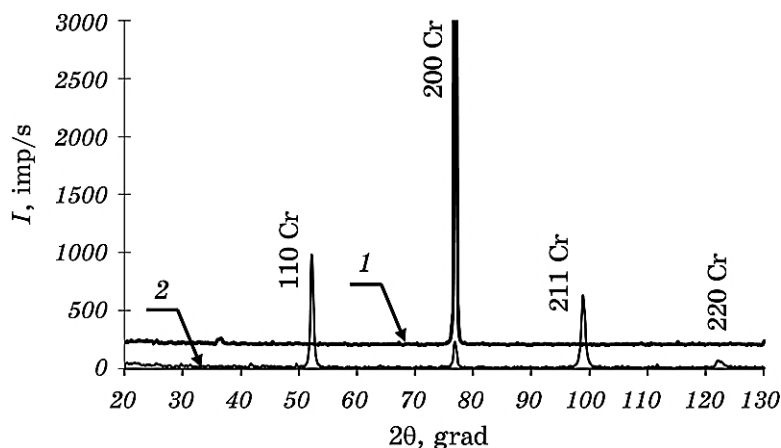


Fig. 7. Diffraction patterns of chromium coatings obtained in a steady-state deposition mode (1) and in a pulsed mode (2) in an argon gas environment.

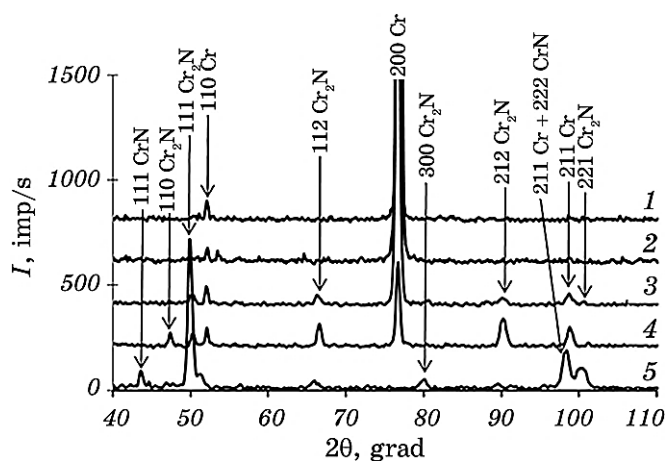


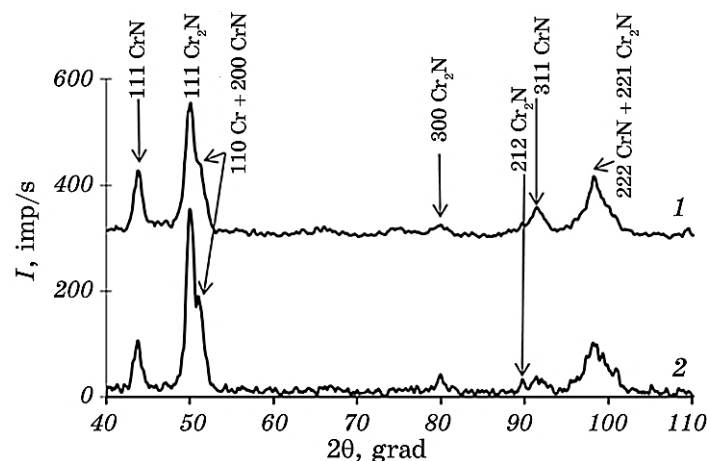
Fig. 8. Diffraction patterns of chromium coatings obtained in a steady-state deposition mode in a gas environment with different percentages of nitrogen: 20%N₂80%Ar (1); 40%N₂60%Ar (2); 60%N₂40%Ar (3); 80%N₂20%Ar (4); 100%N₂ (5).

increase for the amount of chromium nitride Cr₂N, reaching its maximum amount at content of 100% N₂ in the chamber (Table 1). Chromium nitride CrN is formed in a steady-state mode in a small amount (up to 8%) only at the maximum nitrogen content in the chamber.

The pulsed deposition mode intensifies the process of chromium modification with nitrogen. Figure 9 shows the diffraction patterns of chromium coatings modified with nitrogen in the pulsed mode at

TABLE 1. Microstructure and phase composition of chromium coatings under different deposition conditions.

Deposition conditions	Composition of the gas environment	Cr ₂ N, %	CrN, %	<i>a</i> , Å	<i>L</i> , nm	<i>D</i> × 10 ¹⁰ , cm ⁻²
Steady-state mode	100%Ar	—	—	2.8985	1600	4.04
	20%N ₂ 80%Ar	0.5	—	2.8825	1100	7.72
Modification with nitrogen in steady-state mode	40%N ₂ 60%Ar	0.5	—	2.8825	1050	8.64
	60%N ₂ 40%Ar	8	—	2.8870	950	9.77
	80%N ₂ 20%Ar	34	—	2.8889	920	10.59
	100%N ₂	78	8	2.8988	570	38.5
Modification in pulse mode	100%Ar	—	—	2.8879	1340	9.51
	60%N ₂ 40%Ar	55	29	2.8880	700	34.0
	80%N ₂ 20%Ar	70	25	2.8988	520	39

**Fig. 9.** Diffraction patterns of nitrogen-modified chromium coatings in pulse mode: 60%N₂40%Ar (1), 80%N₂20%Ar (2).

60%N₂40%Ar and 80%N₂20%Ar in the chamber. The use of the pulsed mode with a content of 60%N₂40%Ar in the chamber leads to a significant increase for the amount of Cr₂N and CrN nitrides compared to the steady-state mode (Table 1). This effect can be associated with the action of a short pulse with the supply a high bias voltage, which enhances the ionization of nitrogen atoms and intensifies the formation of nitrides.

The studies of the coating microstructure showed that the nitrogen-modified chromium coatings become more fine-crystalline and more

defective. The results of calculations of the lattice parameter, the sizes of the CSRs and the dislocation density are presented in Table 1. The values of the lattice parameter of chromium obtained in a gas environment with the addition of nitrogen are smaller than those for a pure chromium coating that indicates compressing stresses. With an increase in the percentage of nitrogen in the chamber, the lattice parameter increases and reaches its highest value at 100% N₂ in the chamber in the steady-state mode and 80%N₂20%Ar in the pulsed mode, which may indicate the introduction of nitrogen atoms into the chromium lattice. An increase in the percentage of nitrogen in the gas environment of the chamber leads to a decrease in the size of the CSRs and an increase in the dislocation density.

Thus, by varying the nitrogen content in the nitrogen-argon gas mixture in the chamber and the deposition modes, it is possible to control the structure and phase composition of chromium coatings during their arc-PVD.

4. CONCLUSIONS

The work establishes the patterns of formation of the structure and phase composition of chromium coatings obtained by arc-PVD in an argon-nitrogen gas environment, on the basis of which the following conclusions can be made:

- the coating obtained in the steady-state deposition mode in an argon environment has a columnar coarse-crystalline structure with a CSRs size of 1600 nm and an explicit texture [200];
- the use of a pulsed deposition mode in an argon environment significantly weakens the degree of texture, reduces the CSRs size to 1340 nm and disrupts the columnar growth of the coating with a transition to a columnar-globular structure;
- an increase in the percentage of nitrogen in the gas mixture in the chamber under steady-state deposition conditions helps to reduce the CSRs size from 1100 nm to 920 nm, increase the density of dislocations, and change the structure of the coating from columnar to globular;
- modification of the chromium coating with nitrogen in the steady-state deposition mode leads to the formation of 8% Cr₂N at a content of 60%N₂40%Ar in the chamber to 34% Cr₂N at 80%N₂20%Ar in the chamber;
- the use of pulse mode during modification with nitrogen helps to increase the amount of nitrides in the coating from 55% Cr₂N and 29% CrN with a content of 60%N₂40%Ar in the chamber to 70% Cr₂N and 25% CrN with 80%N₂20%Ar in the chamber;
- coatings obtained in an environment of 100% N₂ have unsatisfactory quality: non-uniform in thickness, brittle, delaminated in planes par-

allel to the substrate.

The established patterns of the structure and phase composition of modified chromium coatings can be used to predict their mechanical properties and to build models of structural zones.

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AUTHORS' CONTRIBUTIONS

E. P. Shtapenko supervised the project, developed the concept, formulated the main objective of the study, and performed the overall analysis of the results. V. M. Nadтока conducted the literature review, collected data on the methods of preparation and physical properties of modified chromium coatings, supervised the progress of the work, and wrote Sec. 1 ‘Introduction’ of the manuscript. M. V. Krayev planned the experimental part of the study, developed the vacuum-arc deposition regimes for nitrogen-modified chromium coatings, and wrote Sec. 2 ‘Materials and Methods’ of the manuscript. V. S. Krayeva carried out the experimental investigation and analysis of the structure and phase composition of the obtained coatings and wrote Sec. 3 ‘Results and Discussion’ of the manuscript.

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