

## Structure and Properties of Vacuum-arc Coatings of Chromium and Its Nitrides Obtained under the Action of Constant and Pulse High-voltage Bias Potential

O.V. Sobol<sup>1,\*</sup>, A.A. Postelnyk<sup>1</sup>, R.P. Mygushchenko<sup>1</sup>, Ubeidulla F. Al-Qawabeha<sup>2</sup>, Taha A. Tabaza<sup>3</sup>, Safwan M. Al-Qawabah<sup>3</sup>, V.F. Gorban<sup>4</sup>, V.A. Stolbovoy<sup>5</sup>

<sup>1</sup> National Technical University «Kharkiv Polytechnic Institute», 2, Kyrpychov Str., 61002 Kharkiv, Ukraine

<sup>2</sup> Tafila Technical University, At-Tafilah, P O Box 179, 66110 Tafila, Jordan

<sup>3</sup> Al-Zaytoonah University Queen Alia, 594, Airport Str., 11733 Amman, Jordan

<sup>4</sup> Frantsevich Institute for Problems of Materials Science, 3, Krzhizhanovsky Str., 03142 Kyiv-142, Ukraine

<sup>5</sup> National Science Center «Kharkov Institute of Physics and Technology», 1, Akademicheskaya Str., 61108 Kharkiv, Ukraine

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To reveal the regularities of structural engineering of vacuum-arc coatings based on chromium and its nitrides, the influence of the main physicochemical factors (the pressure of the nitrogen atmosphere and the bias potential) in the formation of coatings was studied. It was discovered that during the deposition of chromium coatings the formation of the texture axis [100], as well as the macrodeformation of compression is happening. The supply of a high-voltage negative pulse potential to the substrate increases the mobility of the deposited atoms and leads to relaxation of the compression deformation. As the pressure increases from  $2 \cdot 10^{-5}$  Torr to  $4.8 \cdot 10^{-3}$  Torr, the phase composition of the coatings changes: Cr (JCPDS 06-0694)  $\rightarrow$  Cr<sub>2</sub>N (JCPDS 35-0803)  $\rightarrow$  CrN (JCPDS 11-0065). The supply of high-voltage pulses leads to the formation of a texture of crystallites with parallel growth surfaces planes having  $d \approx 0.14$  nm. The structure obtained by pulsed high-voltage action makes it possible to increase the hardness of the coating to 32 GPa and reduce the friction coefficient to 0.32 in the "chromium nitride-steel" system and to 0.11 in the "chromium nitride-diamond" system.

The results obtained are explained from the viewpoint of increasing the mobility of atoms and the formation of cascades of displacements when using an additional high-voltage potential in the pulse form during the deposition of chromium-based coatings.

**Keywords:** Vacuum arc, Cr, CrN, Pressure, Bias potential, Pulse potential, Phase composition, Structure, Hardness, Friction coefficient.

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### 1. INTRODUCTION

It is known that an increase of the ionic component in the flow of deposited particles leads to a significant change in the structure and properties of the coatings obtained [1]. It was found that the functional characteristics of the coatings depend significantly on the phase composition [2], the structure of the coatings [3], the degree of texturization [4], and the stress-strain state [5]. The use of nitrogen ions as charged particles made it possible to form coatings of nitrides, the performance characteristics of which significantly exceeded the analogous properties of metals [6].

Coatings based on chromium nitride are of interest, both for basic research and for applied use, taking into account the high functional properties in nitride chromium. Thus, CrN coatings is one of the widely used as protective in such areas as automotive, aerospace industry [7], for die and mould, mechanical components, and artificial joints to increase their service life due to their excellent oxidation resistance, corrosion resistance, low friction coefficient, and high wear resistance [8-12].

At this point, the properties of Cr-N coatings depend in a determined manner on their structure, and, consequently, on the method for their preparation.

Most practical applications have found methods of DC magnetron sputtering [11], high power pulse magnetron sputtering [13] and cathodic vacuum arc [14]. With these technological methods, the formation of coatings occurs under very nonequilibrium conditions, which greatly expands the concentration and temperature regions of existence of phases with a cubic lattice [15]. For chromium nitride, such a stable phase in a wide concentration range is the CrN phase of the structural type of NaCl [16, 17]. Although according to the data given in [18], the energies for the formation of Cr<sub>2</sub>N and CrN phases differ slightly:  $-122.88$  kJ/mol and  $-123.98$  kJ/mol (at 400 °C and constant nitrogen pressure). In addition, chromium has a significantly lower affinity for nitrogen than, for example, titanium. In this regard, the phase composition of the obtained chromium nitride is very sensitive to the physical and technological conditions of deposition.

Therefore, the aim of this work was to study the influence of the basic physical and technological parameters (the pressure of the nitrogen atmosphere and the bias potential) under the vacuum arc method of obtaining coatings on the phase composition, structure, and physicochemical properties of coatings based on chromium and its nitrides.

\* [sool@kpi.kharkov.ua](mailto:sool@kpi.kharkov.ua)

## 2. EXPERIMENTAL DETAILS

The coatings were deposited in the modernized vacuum-arc unit "Bulat-6" [1]. Cr was used as the cathode material with a purity of 99.95 %. The active gas is nitrogen (99.9 %). To obtain chromium coatings, the pressure in the vacuum chamber did not exceed  $2 \cdot 10^{-5}$  Torr during deposition, and when the coatings of chromium nitride were deposited, the pressure of the nitrogen atmosphere was  $P_N = (3.5 \cdot 10^{-4} \dots 4.8 \cdot 10^{-3})$  Torr. During deposition, a constant negative potential was applied to the substrate with a value of  $U_b = -30, -60, -110$  and  $-170$  V. In order to avoid overheating of the substrate and a substantial increase in the mobility of atoms in deposition [19], a high potential  $U_{HVP}$  with an amplitude of 2 kV in a pulsed mode with a frequency of 7 kHz and a duration of 10  $\mu$ s (corresponding to  $\sim 7\%$  of the total application time) was applied. The duration of the deposition process was 1 to 2 hours. The plates were used as substrates and were made of stainless steel 12X18N10T with dimensions  $18 \times 18 \times 2$  mm and copper foils.

The phase composition, structure, and substructural characteristics were studied by the X-ray diffractometry (DRON-4) using Cu-K $\alpha$  radiation. To monochromatize the detected radiation, a graphite monochromator was used, which was installed in a secondary beam (in front of the detector). The phase composition, structure, and substructure characteristics were studied using traditional x-ray diffractometry techniques by analyzing the position, intensity, and shape of the diffraction reflection profiles. To decode the diffractograms, the tables of the international diffraction data center Powder Diffraction File were used.

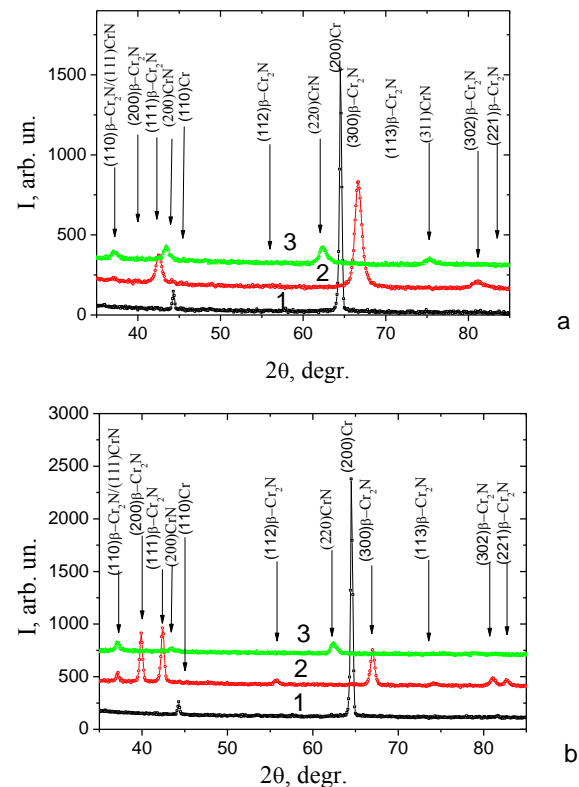
Microindentation was carried out at the «Micron-gamma» unit with a load up to  $F = 0.5$  N with a Berkovich diamond pyramid with an angle of sharpening  $65^\circ$ , with automatic loading and unloading for 30 seconds [20]. The study of the wear resistance characteristics was carried out on the «Micron-friction» device by rotating a diamond indenter with a radius of curvature of approximately 500  $\mu$ m along the coating circle. In the automatic mode, the load level ( $P$ ) and the frictional force between the diamond surface and the coating ( $F$ ) were recorded. The friction coefficient ( $f$ ) was determined as a result of the ratio of the frictional force to the load.

## 3. RESULTS AND THEIR DISCUSSION

Let us consider the results of the influence of two main parameters responsible for the energy state of the deposited particles (negative bias potential in a constant and high-voltage pulse forms) on the structural-phase changes in the coatings obtained by vacuum-arc evaporation of pure chromium in high vacuum (at a pressure of  $2 \cdot 10^{-5}$  Torr). The deposited in such way coatings are effectively used for protection of bearings of heavy-loaded elements of gas turbine aggregates and other elements [21].

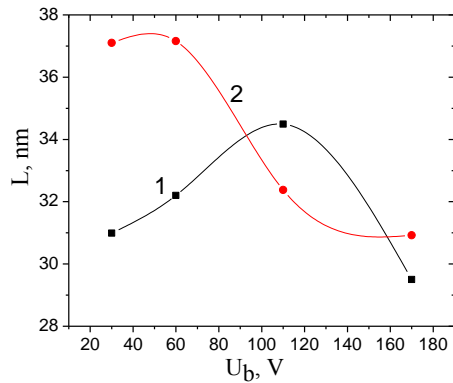
In the way of revealed regularity by the analysis of the diffraction spectra of the coatings (Fig. 1), it can be noted that at a pressure of  $2 \cdot 10^{-5}$  Torr for the entire  $U_b$  interval, the single-phase state of Cr with the volume-centered, bcc, crystal lattice (JCPDS 06-0694) is

formed. All coatings are strongly textured with the axial texture axis [100]. With the greatest displacement potentials, the formation of the second texture axis [110] is revealed and thus the formation of a biaxial state (Fig. 1 a, b, spectrum 1). The period of the lattice of coatings obtained at low  $U_b$  was close to the tabulated 0.2883 nm, and at a large  $U_b$  it increased to 0.2886 nm. In a single-element and single-phase material, the cause of such a change can be the formation of a stress-strain compression state [5]. Evaluation of the macrodeformation gives a compression value of  $-0.43\%$ . It should be noted that, along with  $U_b$ , the supply of a high-voltage bias potential in pulse form (amplitude 2 kV, duration 10  $\mu$ s, which is 7 % of the time from the total exposure time) does not qualitatively change the general form of the spectra. However, there is no increase in the period for large  $U_b$  in this case. The results obtained show that as a result of high-voltage pulse action (this leads to an increase in energy and the formation of cascades in the deposition of particles), the mobility of the particles increases, which leads to relaxation of compression deformation.



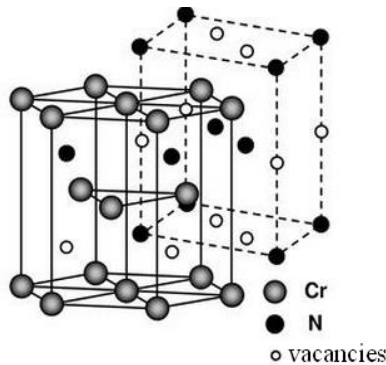
**Fig. 1** – Areas of diffraction spectra of coatings obtained at  $U_b = -30$  V (a) and  $U_b = -110$  V (b) at the working pressure of the nitrogen atmosphere  $P_N$ , Torr: 1 –  $2 \cdot 10^{-5}$ , 2 –  $5 \cdot 10^{-4}$ , 3 –  $1.3 \cdot 10^{-3}$

In this case, as was established by analyzing the width of the diffraction profiles [3], the crystallite size in the coatings with increasing  $U_b$  tends to increase to  $U_b = -110$  V (without  $U_{HVP}$  feed) with a further drop to an average size of about 30 nm at the largest  $U_b = -170$  V (Fig. 2, spectrum 1). At small  $U_b$ , under the conditions of  $U_{HVP}$  action, the average crystallites size is much larger (up to 38 nm), which may be due to greater mobility of the atoms deposited under the action of  $U_{HVP}$ .



**Fig. 2** – The change in the average size of crystallites in Cr coatings obtained at a pressure of  $2 \cdot 10^{-5}$  Torr and different constant bias potentials on the substrate ( $U_b$ ) without additional high-voltage pulse action (1) and with additional pulse action with an amplitude of 2 kV and a frequency of 7 kHz (2)

With increasing pressure ( $P_N$ ), nitride phases are formed. At  $P_N = 3.5 \cdot 10^{-4}$  Torr, the main nitride phase is  $\beta$ -Cr<sub>2</sub>N (JCPDS 35-0803) with a hexagonal type of crystal lattice (Fig. 3). With increasing  $U_b$ , a texture of the crystallites with a texture plane (111) is formed (Fig. 1, a, b, spectrum 2).

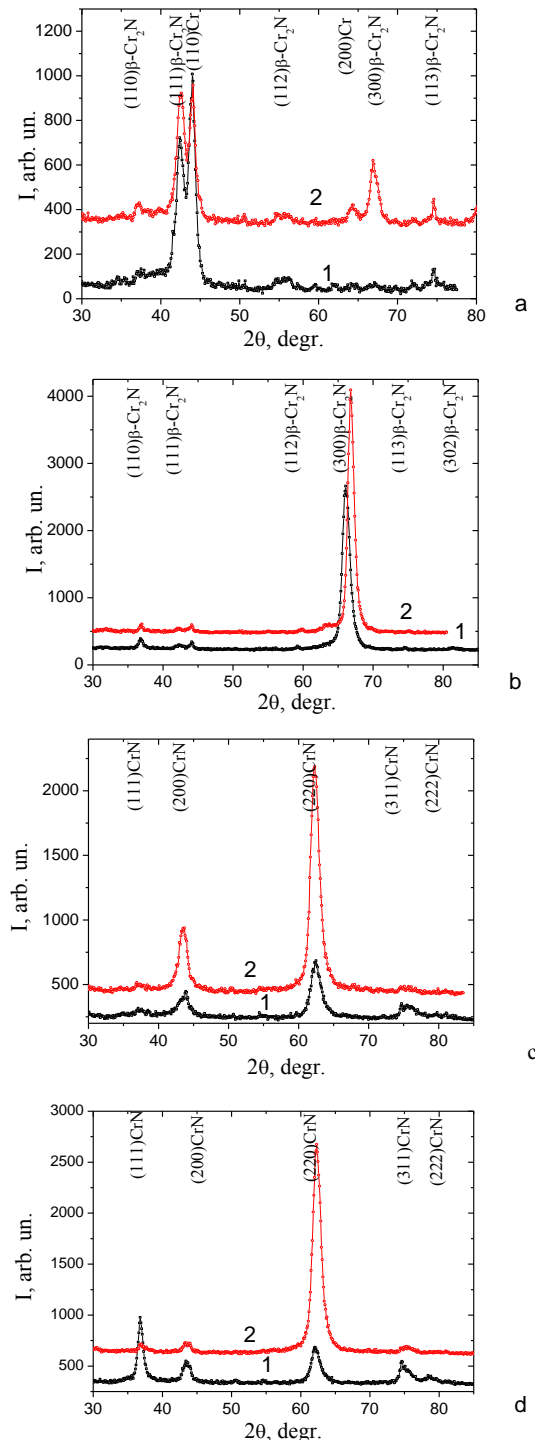


**Fig. 3** – The arrangement scheme of atoms and vacancies in the hexagonal crystal lattice of the  $\beta$ -Cr<sub>2</sub>N phase

At a pressure of  $P_N = 1.3 \cdot 10^{-3}$  Torr, the formation of the CrN phase occurs (JCPDS 11-0065).

Comparison of the diffraction spectra for different  $P_N$  coatings obtained under the action of a large  $U_b = -110$  V without and with  $U_{HVP}$  (Figure 4) showed that at the lowest pressure ( $3.5 \cdot 10^{-4}$  Torr) a two-phase state forms. The crystallites of the phases are textured with the most densely packed planes parallel to the growth plane: (110) Cr and (111)  $\beta$ -Cr<sub>2</sub>N (Fig. 4a). Note that the  $U_{HVP}$  supply additionally stimulates the formation of (300)  $\beta$ -Cr<sub>2</sub>N texture with a characteristic interplanar spacing of 0.14 nm (spectrum 2 in Fig. 4a). When the pressure is raised to  $7.5 \cdot 10^{-4}$  Torr, the texture with the plane (300)  $\beta$ -Cr<sub>2</sub>N becomes the main one, and the coatings are formed practically in a single-phase state. When the pressure is increased to  $1.8 \cdot 10^{-3}$  Torr (Fig. 4c), the already almost single-phase state is formed on the basis of the CrN phase with the cubic lattice and with the texture plane (220). It is interesting to note that the interplanar distance parallel to the plane of the surface is also about 0.14 nm. At the maximum pressure of  $4.8 \cdot 10^{-3}$  Torr (Fig. 4d), under

the action of  $U_{HVP}$ , the texture (220) is amplified (spectrum 2), and without  $U_{HVP}$  supply, coatings are formed with the standard texture (111) for FCC lattice at these pressures [4].



**Fig. 4** – Areas of diffraction spectra of chromium nitride coatings obtained at  $U_b = -110$  V without (1) and with  $U_{HVP}$  (2) at different  $P_N$ , Torr: a –  $3.5 \cdot 10^{-4}$ , b –  $7.5 \cdot 10^{-4}$ ; c –  $1.8 \cdot 10^{-3}$ ; d –  $4.8 \cdot 10^{-3}$

At a sub-structural level under similar formation conditions, the crystallite sizes in the coatings obtained without  $U_{HVP}$  are 10-15 % less than in coatings obtained with  $U_{HVP}$ . At the maximum pressure, their size

reaches 14 nm, and the value of the microdeformation is  $\langle \epsilon \rangle \approx 0.26\%$ .

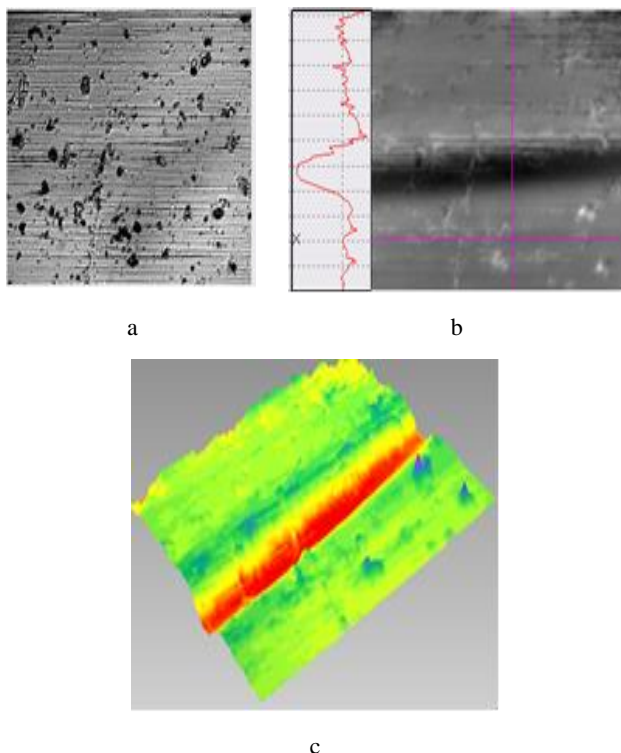
The most universal characteristic that determines the mechanical properties of the coating is hardness. The results of microindentation of chromium nitride coatings obtained at different  $P_N$  are given in Table 1.

**Table 1** – Physicomechanical properties of chromium-based nitride coatings obtained by various technological parameters

$U_b$ , V	$U_{HVP}$ , kV	$P$ , mTorr	$H$ , GPa	$H/E^*$	$E^*$ , GPa	$E$ , GPa
-110	-	4,8	20	0.092	216	256
-110	-	1,8	25.5	0.117	216	255
-110	-	0,75	25.5	0.103	247	302
-110	-	0,35	20.5	0.097	209	246
-110	-2	4,8	27	0.114	236	286
-110	-2	1,8	29	0.111	260	324
-110	-2	0,75	32	0.112	285	364
-110	-2	0,35	28	0.090	308	405

It can be seen that the use of the pulse potential  $U_{HVP}$  leads (at comparable production conditions) to a significant increase in hardness. The highest values of hardness are inherent in coatings obtained in the interval  $P_N = (7.5 \cdot 10^{-4} \dots 1.8 \cdot 10^{-3})$  Torr.

It should also be noted that for the hardest coatings, the ratio  $H/E^* \approx 0.11$  ( $H$  is hardness,  $E^*$  is the reduced elastic modulus, Table 1).

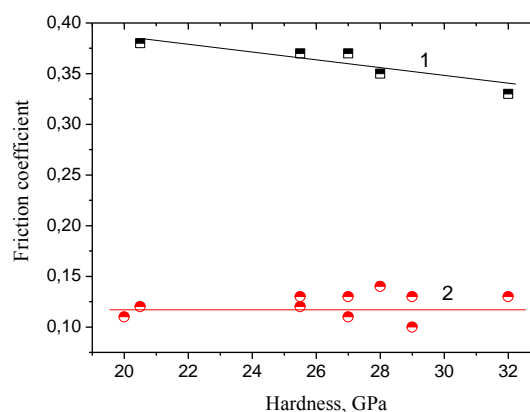


**Fig. 5** – Photographs of the friction surface (a), the groove depth scanning curve (b) and the 3D groove profile and the friction surface (c) of the chromium nitride samples

The second important mechanical characteristic that determines the performance of the coating is its wear resistance. As a rule, coatings with the greatest possible hardness, adhesion and minimum friction coefficient have the smallest wear [22]. Thus, one of the indicators of wear resistance is the friction coefficient. To determine it, the method of rotation of the loaded indenter was used in the work.

Figure 5 shows the surface photographs and the 3D profile of the groove and the friction surface.

Tribological tests were carried out for two types of pairs "chromium nitride - steel ShKh" and "chromium nitride – diamond". Figure 6 shows the results of such tests, made depending on the hardness of the coatings. It can be seen that, for chromium-based nitride coatings, there is a tendency to reduce the friction coefficient with increasing stiffness in pairing with steel ShKh (Fig. 6, curve 1) and stability of the friction coefficient in pair with diamond (Fig. 6, curve 2). In absolute magnitude, in the first case, the friction coefficient is 0.32, and in the second, about 0.12.



**Fig. 6** – Dependence of the friction coefficient of the coating on the basis of chromium nitride in pair with steel ShKh (1) and diamond (2)

The obtained test results are quite predictable. The low friction coefficient in the case of a pair of "chromium nitride – diamond" is determined by the possibility of forming on the contacting surfaces of the carbon layer (serves as a solid lubricant). In the case of a pair of "chromium nitride – steel ShKh", such a lubricant is not formed, the friction meets the rigid conditions of solid-state contact.

#### 4. CONCLUSIONS

Thus, the X-ray structural analysis of coatings in combination with the study of tribological characteristics (tribology can be defined as the science of surface layers strength of solid materials with their relative displacement) showed that in the hardest and most wear-resistant coatings of chromium nitride (with the lowest friction coefficient) CrN crystallites with plane orientation (220) are parallel to the working surface.

## Структура і властивості вакуумно-дугових покриттів хрому і його нітридів, отриманих в умовах дії постійного і імпульсного високовольтного потенціалів зсуву

О.В. Соболев<sup>1</sup>, Г.О. Постельник<sup>1</sup>, Р.П. Мигущенко<sup>1</sup>, Ubeidulla F. Al-Qawabeha<sup>2</sup>, Taha A. Tabaza<sup>3</sup>, Safwan M. Al-Qawabah<sup>3</sup>, В.Ф. Горбань<sup>4</sup>, В.А. Столбовий<sup>5</sup>

<sup>1</sup> Національний технічний університет «Харківський політехнічний інститут», вул. Кирпичова, 2, 61002 Харків, Україна

<sup>2</sup> Tafila Technical University, At-Tafilah, P O Box 179, 66110 Tafila, Jordan

<sup>3</sup> Al-Zaytoonah University Queen Alia, 594, Airport Str., 11733 Amman, Jordan

<sup>4</sup> Інститут проблем матеріалознавства ім. Францевича, вул. Кржижановського, 3, 03142 Київ-142, Україна

<sup>5</sup> Національний науковий центр «Харківський фізико-технічний інститут», вул. Академічна, 1, 61108 Харків, Україна

Для виявлення закономірностей структурної інженерії вакуумно-дугових покриттів на основі хрому та його нітридів досліджено вплив основних фізико-технологічних факторів (тиск азотної атмосфери і потенціал зсуву) при формуванні покриттів. Встановлено, що при осадженні покриттів хрому відбувається формування: осі текстури [100], а також макродеформацій стиснення. Подача високовольтного негативного імпульсного потенціалу на підкладку підвищує рухливість атомів, що осаджуються і призводить до релаксації деформації стиснення. Зі збільшенням тиску від  $2 \cdot 10^{-5}$  Торр до  $4,8 \cdot 10^{-3}$  Торр фазовий склад покриттів змінюється: Cr (JCPDS 06-0694)  $\rightarrow$  Cr<sub>2</sub>N (JCPDS 35-0803)  $\rightarrow$  CrN (JCPDS 11-0065). Подача високовольтних імпульсів призводить до формування текстури кристалитів з паралельними поверхні зростання площинами які мають  $d \approx 0.14$  нм. Одержана при імпульсному високовольтному впливі структура дозволяє підвищити твердість покриття до 32 ГПа і знизити коефіцієнт тертя до 0.32 в системі «нітрид хрому – сталь» і до 0.11 в системі «нітрид хрому – алмаз».

Одержані результати пояснені з позиції підвищення рухливості атомів і утворення каскадів зміщення при використанні в процесі осадження покриттів на основі хрому додаткового високовольтного потенціалу в імпульсній формі.

**Ключові слова:** Вакуумна дуга, Cr, CrN, Тиск, Потенціал зсуву, Імпульсний високовольтний потенціал, Фазовий склад, Структура, Твердість, Коефіцієнт тертя.

## Структура и свойства вакуумно дуговых покрытий хрома и его нитридов, полученных в условиях действия постоянного и импульсного высоковольтного потенциалов смещения

О.В. Соболев<sup>1</sup>, А.А. Постельник<sup>1</sup>, Р.П. Мигущенко<sup>1</sup>, Ubeidulla F. Al-Qawabeha<sup>2</sup>, Taha A. Tabaza<sup>3</sup>, Safwan M. Al-Qawabah<sup>3</sup>, В.Ф. Горбань<sup>4</sup>, В.А. Столбовой<sup>5</sup>

<sup>1</sup> Национальный технический университет «Харьковский политехнический институт», ул. Кирпичова, 2, 61002 Харьков, Украина

<sup>2</sup> Tafila Technical University, At-Tafilah, P O Box 179, 66110 Tafila, Jordan

<sup>3</sup> Al-Zaytoonah University Queen Alia, 594, Airport Str., 11733 Amman, Jordan

<sup>4</sup> Институт проблем материаловедения им. Францевича, ул. Кржижановского, 3, 03142, Киев-142, Украина

<sup>5</sup> Национальный научный центр «Харьковский физико-технический институт», ул. Академическая, 1, 61108 Харьков, Украина

Для установления закономерностей структурной инженерии вакуумно-дуговых покрытий на основе хрома и его нитридов изучено влияние основных физико-технологических факторов (давление азотной атмосферы и потенциал смещения) при формировании покрытий. Установлено, что при осаджении покрытий хрома происходит формирование оси текстуры [100], а также макродеформации сжатия. Подача высоковольтного отрицательного импульсного потенциала на подложку повышает подвижность осаждаемых атомов и приводит к релаксации деформации сжатия. С увеличением давления от  $2 \cdot 10^{-5}$  Торр до  $4,8 \cdot 10^{-3}$  Торр фазовый состав покрытий изменяется: Cr (JCPDS 06-0694)  $\rightarrow$  Cr<sub>2</sub>N (JCPDS 35-0803)  $\rightarrow$  CrN (JCPDS 11-0065). Подача высоковольтных импульсов приводит к формированию текстуры кристаллитов с параллельными поверхности роста плоскостями имеющими  $d \approx 0.14$  нм. Полученная при импульсном высоковольтном воздействии структура позволяет повысить твердость покрытия до 32 ГПа и понизить коэффициент трения до 0.32 в системе «нитрид хрома – сталь» и до 0.11 в системе «нитрид хрома – алмаз».

Полученные результаты объяснены с позиции повышения подвижности атомов и образования каскадов смещений при использовании в процессе осаджения покрытий на основе хрома дополнительного высоковольтного потенциала в импульсной форме.

**Ключевые слова:** Вакуумная дуга, Cr, CrN, Давление, Потенциал смещения, Импульсний високовольтний потенціал, Фазовий склад, Структура, Твердость, Коэффициент трения.

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