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STUDY OF ADVANCED NANOSCALE ZrN/CrN MULTILAYER COATINGS

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The scientific interest in the investigation of nitride composites as protecting materials in tool and machining industries intensively increases. The good oxidation resistance of CrN single-layer films and high melting point, good chemical and thermal resistance of ZrN compound are motive factors for designing of multilayer composites composed of these metal nitrides. The suggested advantages of ZrN/CrN multilayer coatings as structural materials are the high-temperature resistance, high density and extreme hardness compared to the metal-nitride systems. Experimental ZrN/CrN multilayer coatings were deposited on AISI 321 steel substrates by using a cathodic arc evaporation device equipped with two high-purity metal Cr and Zr targets. Structural, chemical and morphological characteristics together with mechanical properties of multilayer composites were analyzed by X-ray diffraction, scanning electron microscopy, energy-dispersive X-ray spectroscopy and Vickers hardness tester. SEM analysis revealed an increase of roughness and concentration of the droplets on the surface of the coatings when negative bias potential decreased to -70 V. The results of data obtained from the X-ray analysis showed (200) and (111) plane for ZrN and Cr₂N phases as the most intense. The peak positions of ZrN were shifted towards lower diffraction angles comparing with bulk values and indicated a decrease of the inter-planar distance and formation of compressive stresses. The calculated lattice strain values in the ZrN were higher than those of the CrN, indicated a greater presence of dislocations and defects in the lattice of ZrN. The averaged crystallite sizes in ZrN and CrN layers were 11-14 and 7-12 nm, respectively. The maximum value of the Vickers microhardness was found to be 6600HV0.01 that is 2.1 and 1.8 times greater than the corresponding values of binary CrN and ZrN coatings.

KEYWORDS: nitrides, cathodic arc deposition, microstructure, elemental composition, structural-phase state.

The extension of the operational life of industrial equipment, components of installations, cutting, drilling and other machining tools stays a relevant task of science and technology of materials engineering till present. Moreover, nowadays at the stage of an enhanced economy of resources and in going to energy-saving technologies, this issue becomes even more acute. While functioning, the product surface layers undergo the strongest loading, physical, chemical and thermal effects. One of the ways to protect and improve various material properties is to modify it due to deposition of nanostructured thin coatings on its surface.

Cathodic-arc technique is a multipurpose method of coating deposition since the resulted products gain wide industrial distribution because of their suitability for various functional purposes [1, 2]. Transition metal nitride coatings with a thickness of a few microns are one of the most studied and widely used materials. Unfortunately, the possibilities of increasing the hardness and plasticity of the surface layer during the deposition of simple nitrides are practically depleted, since at mid-temperature range mononitrides initiated to be thermally unstable [3-5].

Recently, nanoscale coatings of a complex elemental and phase composition realized through the multilayer coating concept are of considerable interest, since combinations of various elements make it possible to use the best properties of two or several metals and their nitrides [6, 7].

This paper describes the effect of deposition parameters on the structure, elemental and phase composition of multilayer coatings ZrN/CrN, as well as their mechanical characteristics.

EXPERIMENTAL DETAILS

The ZrN/CrN multilayers were fabricated by the cathodic-arc method in a Bulat 6 deposition system [8], which composed of a vacuum chamber, vacuum pumping system, nitrogen supply system that worked at pressure between 10⁻⁵ to 10⁻² Pa, arc power supplies that produced the current from 50 to 200 A, substrate power supply that ensured constant negative bias voltage and automatic rotation system for substrate holder. Within the working chamber, there was an electrode formed by an anode, where the steel substrates were placed, and cathodes with the Zr and Cr targets. Metal targets were in the opposite positions and the substrates were mounted on two sides of a rotating substrate holder between the two targets. Experimental coatings were deposited by alternately rotating the substrates between Cr and Zr targets. The arc current of 100 A was constantly applied for all samples. When the first multilayer ZrN/CrN film started to produce, the chamber was filled with nitrogen at a pressure of 0.03 Pa. For other samples of the coatings, it changed

as the goal of this investigation was to evaluate the influence of deposition conditions on morphology, phase state and mechanical properties of multilayer condensates. Specific details of the deposition process of the multilayer coatings were summarized in Table 1.

The surface and cross-section structure, as well as the chemical composition of the coatings, were analyzed by scanning electron microscopy with energy dispersive X-ray spectroscopy facilities (SEM-EDS) in a FEI Quanta 400 FEG ESEM/EDAX Genesis X4M. For the XRD characterization technique, we used a Panalytical X'Pert Pro MPD diffractometer. Phase identification was realized through the ICDD data. Mechanical properties were evaluated by Vickers microhardness tests in a Struers Duramin-5 using a load of 98 mN (HV0.01).

Table 1.

Deposition conditions of multilayer ZrN/CrN coatings.

Sample number	Arc current I_d , A	Bias voltage U_s , V	Substrate temperature T_s , °C	Nitrogen pressure P_N , Pa	Exposing time t , s	Total deposition time, h	Deposition rate R_d , nm/s	Number of layers
1	100	-70	250	0.04	ZrN:10/CrN:10	1	3.25	354
2				0.43			3.5	
3		-150		0.16			4.5	
4				0.03			2.65	

RESULTS AND DISCUSSION

Morphologic study

The morphology of the PVD-coatings is basically controlled by the process characteristics, i.e. substrate temperature (T_s), working gas pressure (P_N), the negative bias voltage applied to the substrate (U_s) and an arc current (I_d) [9, 10]. During the surface analysis of experimental coatings, it is revealed that micro-relief of the surface is expressed by numerous shallow different-sized depressions due to the growth of crystallites of different sizes. The formation of the droplets with a particle diameter averaged from 2 to 5 microns is observed for all the coatings. Increasing the nitrogen pressure up to 0.43 Pa while the deposition does not introduce any special changes in the surface morphology. However, increasing the energy of the precipitated flow through the bias potential to -200 V significantly reduces the concentration of the droplets on the surface. Probably, with the increase of the substrate bias value, the energetic particles bombardment becomes more intensive and activates the process of cleaning the surface from smaller fractions. Fig. 1a shows the cross-sectional SEM image of the multilayer sample number 4. The heterophase interfaces between ZrN and CrN layers are rather straight and immiscible. The dark contrast layers correspond to the CrN, while lighter ones indicate the binary ZrN. The structure of layers is dense, no boundary porosity or other defects exist.

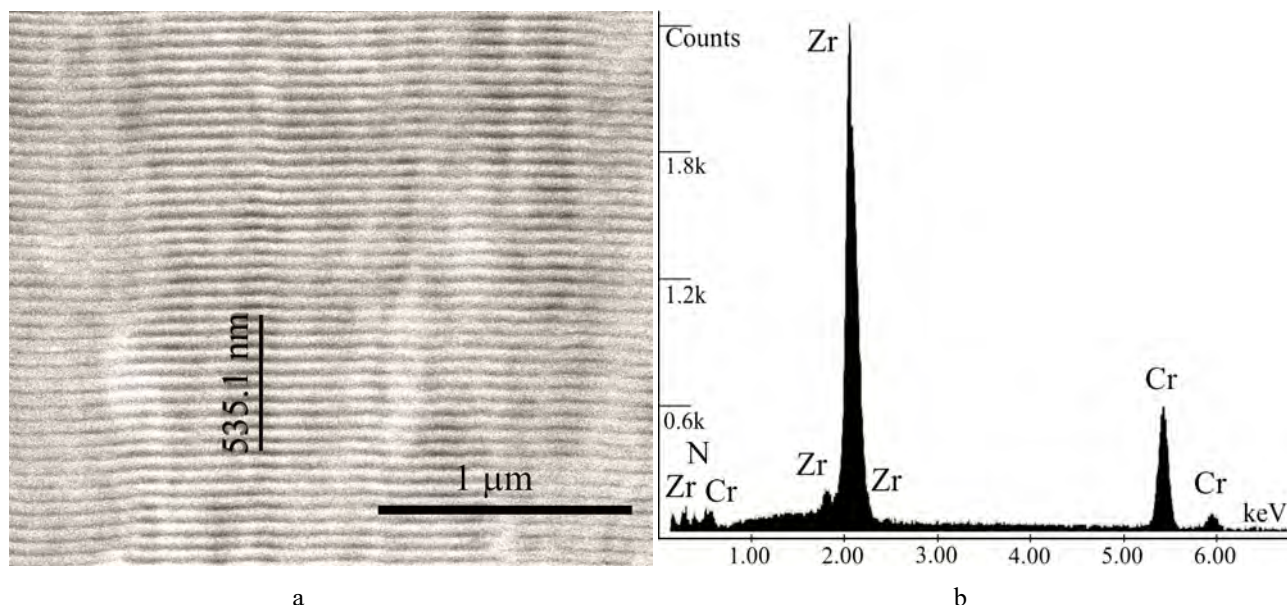


Fig. 1. Cross-sectional SEM image (a) and EDS spectrum (b) of multilayer ZrN/CrN coating number 4

Shown in Fig. 1b EDS spectrum is composed of Zr, Cr and N peaks, that confirms the purity of elemental composition of the coating number 4. The chemical compositions of multilayer ZrN/CrN coatings are generalized in Table 2 together with the total and bilayer thicknesses extracted from SEM images using ImageJ software.

It is seen from data collected via SEM and EDS that obtained coatings have the nanoscale bilayer thickness within the range from 53 to 70 nm and near-stoichiometric or stoichiometric composition as an atomic percentage of nitrogen

changes from 46 to 51. It is obvious, that for experimental coatings there is a tendency to decrease the concentration of metal elements with increasing nitrogen pressure. The lower chromium concentration in comparison with the zirconium concentration is due to the fact that chromium has a lower atomic mass ($\text{Cr} = 51.99 \text{ g/mol}$ vs. $\text{Zr} = 91.22 \text{ g/mol}$), that is, it is a lighter element. The fact is that in the process of coating deposition, when the plasma energy and, accordingly, the intensity of deposition increases, with an increase in P_N , lighter atoms may escape from the surface coating layer, which leads to an increase of another element concentration in the coating.

Table 2.

Characteristics of multilayer ZrN/CrN coatings: average coating thickness, bilayer period and chemical composition.

Sample number	Coating thickness D , μm	Bilayer period λ , nm	Chemical composition, at.%		
			N	Zr	Cr
1	11.5	65	46	30	24
2	12.4	70	50	27	23
3	16.0	90	51	29	20
4	9.4	53	47	35	18

Structural characterization

X-ray diffraction patterns of multilayer ZrN/CrN composites are shown in Fig. 2. As seen in this figure, at all values range of nitrogen pressure there is a clear (200) preferred orientation for ZrN layers and (111) preferred orientation for CrN layers (non-isostructured multilayers). For the coatings deposited at low bias potential, the texture of the ZrN layer with orientation (111) appears at the patterns. This behavior suggests the prerequisites for the cube-on-cube epitaxial growth. Some small contributions of ZrN(220) reflection are identified for samples number 1 and 2, but it disappears for larger values of U_S .

The texture coefficient T_C of (200) plane with respect of (111) plane for ZrN phase and T_C of (111) plane of Cr_2N phase with respect of (111) plane of CrN phase (samples 1 and 2) was calculated by the equation [11]:

$$T_c(hkl) = \frac{I(hkl) / I_0(hkl)}{(1/N) [\sum_N I(hkl) / I_0(hkl)]}, \quad (1)$$

where $I(hkl)$ is the integrated intensity of the diffraction plane calculated using an approximation of the pseudo-Voigt function, $I_0(hkl)$ is the relative intensity of the corresponding plane given in ICDD, and N is the number of reflections.

The evolution of the crystallography texture coefficient in the function of the working gas pressure is clearly observed in Table 3. For the low values of P_N , the texture coefficient values are 2.35 and 1.12 for the ZrN and the Cr_2N , respectively. As the nitrogen pressure increases, the coefficient for both phases lightly and respectively diminishes going to values between 1.51 for the ZrN and 1.07 for the Cr_2N .

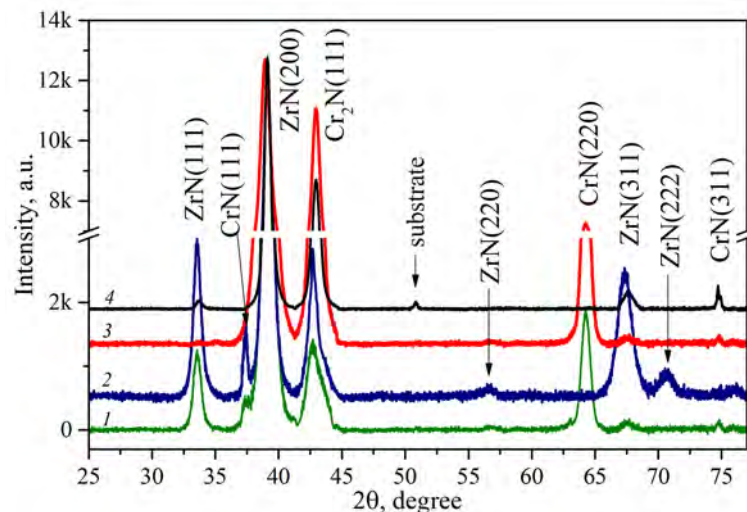


Fig. 2. X-ray diffraction data obtained from multilayer ZrN/CrN coatings

The X-ray method for determining internal stresses is based on the fact that the elastic deformation of the crystal lattice is expressed by the changes in inter-planar distance and diffraction angle in the X-ray patterns. It is necessary to point out one feature of the X-ray method for measuring residual stresses. The X-ray diffraction line is formed as a result of the total reflection from those crystallographic planes for which the Wulff-Bragg condition is satisfied. Thus, all calculations carried out on the basis of measuring the angular positions of X-ray lines provide information on the

stress state of only certain oriented grains of a polycrystalline sample. In this study, the lattice strains ϵ of ZrN and CrN were calculated according to the following equation based on (200)ZrN and (111)Cr₂N planes [12]:

$$\epsilon = \frac{d - d_0}{d_0}, \tag{2}$$

where d_0 is the inter-planar distance of distinct (hkl) planes of the residual stress-free lattice and d is the inter-planar distance of the same planes influenced by residual stress. It is obvious from the Table 3 that the strains of the ZrN is greater than those of the CrN, indicating a larger formation of dislocations and defects in ZrN lattice. These dislocations cause the subdivision of the original crystallites of larger size into domains (small crystallites), producing tensile and compressive strains within diminished crystallites [13].

Table 3.

Values of strain-size parameters calculated from X-ray patterns of ZrN/CrN coatings

Sample number	Investigated phase and plane	Experimental diffraction angle 2θ , deg	Standard diffraction angle $2\theta_0$, deg	Experimental inter-planar distance d , nm	Standard inter-planar distance d_0 , nm	Crystallite size L , nm	Texture coefficient T_c	Lattice strain ϵ , %
1	ZrN (200)	39.10	39.13	0.2305	0.2309	11	2.35	-0.17
	Cr ₂ N(111)	42.77	42.72	0.2111	0.2114	7	1.12	-0.14
2	ZrN(200)	39.12	39.13	0.2302	0.2309	12	1.51	-0.30
	Cr ₂ N(111)	42.77	42.72	0.2112	0.2114	10	1.07	-0.09
3	ZrN(200)	39.10	39.13	0.2305	0.2309	14	2.05	-0.17
	Cr ₂ N(111)	42.79	42.72	0.2112	0.2114	12	-	-0.09
4	ZrN(200)	39.12	39.13	0.2306	0.2309	13	2.67	-0.13
	Cr ₂ N(111)	42.78	42.72	0.2110	0.2114	11	-	-0.18

Peaks obtained from ZrN demonstrate the shift toward lower angles (2θ ranged from 39.10 to 39.12°), that signifies the existence of compressive stresses in ZrN layers. A significant movement of the Cr₂N peaks towards higher angles ($2\theta = 42.77$ and 42.79°) in relation to standard values ($2\theta = 42.72^\circ$) shows the presence of tensile stresses in CrN layers.

Mechanical properties

Hardness values of the multilayer ZrN/CrN coatings measured using the Vickers tip, which is a four-sided pyramid, are presented in Fig. 3. All samples acquire the hardness that overperforms the corresponding value of individual coatings, i.e., CrN and ZrN monolayers. It is observed a hardness-increasing trend as the nitrogen pressure in the vacuum chamber decreases. The highest hardness of 6600HV0.01 is obtained for sample number 2 with stoichiometric composition of 50 at.% N and bilayer period of 70 nm, that is on 51 % and 47 % greater than referent value from ZrN and CrN coatings, respectively.

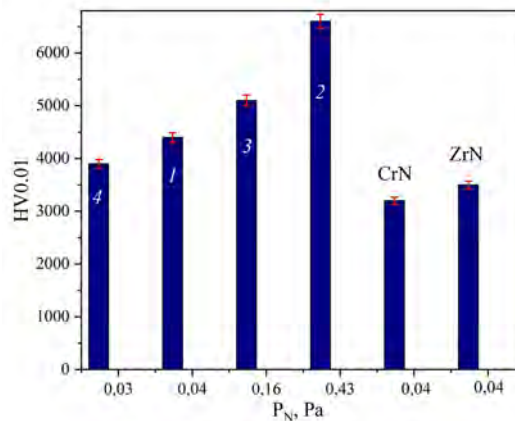


Fig. 3. Variation of microhardness with nitrogen pressure values for the multilayer ZrN/CrN coatings and binary ZrN and CrN coatings.

In general, the increase of the mechanical properties of multilayer coating lies in application of the multilayer concept that possess the following conditions: (i) the presence of many interfaces that blocks the dislocation movement across the interface between ZrN and CrN layers; (ii) coherency strains cause periodical strain-stress fields due to the lattice-mismatch in the multilayer coating; (iii) the formation of the discrete layers in the multilayer system, which observed in the SEM results; (iv) the formation of partial correlation within (111) crystallography direction between ZrN and CrN layers.

CONCLUSIONS

In summary, the morphological and structural-phase characteristics, as well as mechanical properties of multilayer ZrN/CrN coatings grown on stainless steel substrates by the cathodic arc deposition were studied.

It was shown that the surface of the multilayer composited contained droplet particles, which concentration depended on negative bias potential applied to the steel substrate. The total thickness of the obtained films ranged from 9.4 to 16.0 μm , while the bilayer period varied from 53 to 90 nm. The elemental composition of cathodic arc coatings depended on working gas pressure, hence, at high values of P_N formed coatings had the stoichiometric composition (50-51 at.%).





Additionally, different deposition conditions, i.e. nitrogen pressure and substrate bias, promoted the preferred formation of two phases ZrN(200) and Cr₂N(111), but at $U_S = -70$ V the intensive peak of ZrN(111) and reflex of CrN(111) were found on diffraction patterns.

The enhancement in microhardness of multilayer ZrN/CrN coatings was observed for all experimental samples. The maximum value 6600HV0.01 was found for the sample number 2 of stoichiometric composition (51 at.% N) obtained at $P_N = 0.43$ Pa, $U_S = -70$ V. Presented findings prove that multilayer ZrN/CrN coatings are appropriate for use as protective materials due to high hardness. However, the tribological, thermal and oxidation experiments of multilayer ZrN/CrN systems need to be done in order to find out their potential and extend the range of possible applications.

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ДОСЛІДЖЕННЯ ПЕРСПЕКТИВНИХ НАНОРОЗМІРНИХ ZrN/CrN БАГАТОШАРОВИХ ПОКРИТТІВ

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Науковий інтерес до дослідження нітридних композитів в якості захисних матеріалів в інструментальній та обробній промисловостях інтенсивно зростає. Хороша стійкість до окислення одношарових плівок CrN і висока температура плавлення, хороша хімічна і термічна стійкість ZrN є рушійними факторами для створення багатошарових композитів, в яких використовуються нітриди цих перехідних металів. Передбачувані переваги багатошарових покриттів в якості

конструкційних матеріалів – це висока термостійкість, висока щільність їх структури і покращена твердість у порівнянні з системами нітрид-метал. Багат шарові покриття ZrN/CrN осаджувалися на сталеві підкладки AISI 321 за допомогою вакуумно-дугового пристрою, оснащеного двома металевими мішенями Cr і Zr високої чистоти. Структурні, хімічні та морфологічні характеристики, а також механічні властивості багат шарових композитів були проаналізовані за допомогою рентгенівської дифракції, растрової електронної мікроскопії, енергодисперсійної рентгенівської спектроскопії і твердомира, оснащеного пірамідкою Віккерса. РЕМ-аналіз виявив збільшення шорсткості і концентрації крапель на поверхні покриттів, коли негативний потенціал зсуву знижувався до значення в -70 В. Результати даних, отриманих від рентгеноструктурного аналізу, показали, що найбільш інтенсивними є площини (200) і (111) для ZrN і Cr₂N фази, відповідно. Положення піків ZrN були зміщені в бік менших кутів дифракції в порівнянні з об'ємними значеннями і вказували на зменшення міжплощинної відстані і формування стискаючих напружень. Розраховані значення напруження решітки в ZrN вище, ніж у CrN, що свідчить про більшу наявності дислокацій і дефектів в решітці ZrN. Усереднені розміри кристалітів в шарах ZrN і CrN становили 11-14 і 7-12 нм відповідно. Було встановлено, що максимальне значення мікротвердості по Віккерсу становить 6600HV0.01, що в 2,1 і 1,8 рази більше, ніж відповідні значення бінарних покриттів CrN і ZrN.

КЛЮЧОВІ СЛОВА: нітриди, вакуумно-дугове осадження, мікроструктура, елементний склад, структурно-фазовий стан.

ИССЛЕДОВАНИЯ ПЕРСПЕКТИВНЫХ НАНОРАЗМЕРНЫХ ZrN/CrN МНОГОСЛОЙНЫХ ПОКРЫТИЙ

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Научный интерес к исследованию нитридных композитов как защитных материалов в инструментальной и обрабатывающей промышленности интенсивно возрастает. Хорошая стойкость к окислению однослойных пленок CrN и высокая температура плавления, хорошая химическая и термическая стойкость ZrN являются движущими факторами для создания многослойных композитов, в которых используются нитриды этих переходных металлов. Предполагаемые преимущества многослойных покрытий в качестве конструкционных материалов – это высокая термостойкость, высокая плотность их структуры и чрезвычайная твердость по сравнению с системами нитрид-металл. Многослойные покрытия ZrN/CrN наносились на стальные подложки AISI 321 с использованием вакуумно-дугового устройства, оснащенного двумя металлическими мишенями Cr и Zr высокой чистоты. Структурные, химические и морфологические характеристики, а также механические свойства многослойных композитов были проанализированы с помощью рентгеновской дифракции, растровой электронной микроскопии, энергодисперсионной рентгеновской спектроскопии и твердомера, оснащенного пирамидкой Виккерса. РЕМ-анализ выявил увеличение шероховатости и концентрации капель на поверхности покрытий, когда отрицательный потенциал смещения снижался до значения в -70 В. Результаты данных, полученных от рентгеноструктурного анализа, показали, что наиболее интенсивными являются плоскости (200) и (111) для ZrN и Cr₂N фазы, соответственно. Положения пиков ZrN были смещены в сторону меньших углов дифракции по сравнению с об'ємними значеннями і вказували на уменшення міжплоскостного расстояния и присутствие сжимающих напряжений. Рассчитанные значения напряжений решетки в ZrN выше, чем у CrN, что свидетельствует о большем наличии дислокаций и дефектов в решетке ZrN. Усредненные размеры кристаллитов в слоях ZrN и CrN составляли 11-14 и 7-12 нм соответственно. Было установлено, что максимальное значение микротвердости по Виккерсу составляет 6600HV0.01, что в 2,1 и 1,8 раза больше, чем соответствующие значения бинарных покрытий CrN и ZrN.

КЛЮЧЕВЫЕ СЛОВА: нитриды, вакуумно-дуговое осаждение, микроструктура, элементный состав, структурно-фазовое состояние.