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A study of the characteristics of nanostructured valve coatings for operation in aggressive conditions

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Abstract: The object of this study is composite multicomponent nanostructured coatings, which are used to increase the durability of the working surfaces of valves operated at critical facilities in the petrochemical and nuclear industries. The aim of thestudy is to experimentally determine the main characteristics of coatings Ti–TiN–(Ti, Mo, Al)N, Ti–CrN–(Cr, Mo, Al)N, Ti–Zr–ZrN–(Zr, Mo, Al)N obtained by the method of substance condensation by cathode-vacuum-arc deposition with filtration of the microdroplet phase, and to select the most preferable characteristics for shut-off valves. To determine the coefficient of dry friction, tribological tests were carried out, instrumental indentation was used to establish the hardness of the samples, and the roughness was determined by the profile method. It was found that the best values of the studied characteristics, namely, indentation hardness (38.9 GPa) and roughness (average profile deviation is 0.242 μm), according to the test results, are samples coated with Cr–CrN–(Cr, Mo, Al)N ; according to the results of tribological tests, the Cr–CrN–(Cr, Mo, Al)N coating also has the lowest friction coefficient (0.0806). A promising direction for further research may be the development and study of new multicomponent nanocomposite coatings based on high-entropy alloys.

Keywords: nanostructured multicomponent coatings; cathodic-vacuum-arc deposition; stop valves; modulus of elasticity during indentation; hardness; roughness; dry friction coefficient; tests.

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Исследование характеристик наноструктурированных покрытий запорной арматуры для эксплуатации в агрессивных условиях

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Аннотация: Объектом настоящего исследования являются композиционные многокомпонентные наноструктурированные покрытия, которые используют для повышения долговечности рабочих поверхностей запорной арматуры, эксплуатируемой на ответственных объектах нефтехимической и атомной промышленности. Цель настоящей работы заключается в экспериментальном определении основных характеристик покрытий Ti–TiN–(Ti, Mo, Al)N, Ti–CrN–(Cr, Mo, Al)N, Ti–Zr–ZrN–(Zr, Mo, Al)N, полученных методом конденсации вещества катодно-вакуумно-дуговым осаждением с фильтрацией микрокапельной фазы, для выбора наиболее предпочтительных для запорной арматуры. В целях определения коэффициента сухого трения проводили трибологические испытания, для установления твердости образцов использовали инструментальное индентирование, шероховатость определяли профильным методом. Установлено, что наилучшими значениями исследуемых характеристик, а именно твердости при индентировании (38,9 ГПа) и шероховатости (среднее отклонение профиля равно 0,242 мкм) по результатам испытаний, обладают образцы с покрытием Cr–CrN–(Cr, Mo, Al)N; по результатам трибологических испытаний наименьший коэффициент трения (0,0806) также имеет покрытие Cr–CrN–(Cr, Mo, Al)N. Перспективным направлением дальнейших исследований может стать разработка и исследование новых многокомпонентных нанокомпозитных покрытий на основе высокоэнтропийных сплавов.

Ключевые слова: наноструктурированные многокомпонентные покрытия; катодно-вакуумно-дуговые осаждения; запорная арматура; модуль упругости при индентировании; твердость; шероховатость; коэффициент сухого трения; испытания.

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1. Introduction

Shut-off valves are an indispensable component of technical equipment used in the processing and oil and gas industries, as well as nuclear energy [1]. Due to the constant technical improvement combined with an improvement of technical characteristics and expansion of the range of permissible loads, as well as due to the need for special equipment for working in aggressive conditions, solving the problem of ensuring wear resistance and durability of technical tools and equipment remains an urgent scientific and technical task [2].

An increase in durability and wear resistance of products is closely related to an increase in their reliability. The rational choice of materials, the optimal design of friction units and the optimization of the operating conditions of the designed products are of great importance for increasing their service life [3, 4]. To improve the characteristics of friction units, a number of different processing methods are used, such as cutting, heat treatment, surfacing, surface plastic deformation hardening, thermo-mechanical and chemical processing, wear-resistant coatings, detonation, gas-plasma, plasma, electric arc spraying, spraying, etc., as well as surface modification due to the deposition of various functional coatings [5–7].

Thus, a significant increase in wear resistance of loaded friction elements of shut-off valves becomes possible as a result of the development and implementation of new technologies for modifying the surface of materials. To date, a large number of different coatings have been developed, among which multicomponent nanocomposite coatings are not in the last place [8–12].

The development and implementation of new materials is a complex task that requires research to confirm their effectiveness [13–15]. In the case of functional coatings, such experimental studies are often tests for hardness, roughness, and tribology [16–23].

The aim of the research is an experimental study of multicomponent nanocomposite coatings Ti–TiN–(Ti, Mo, Al)N, Ti–CrN–(Cr, Mo, Al)N, Ti–Zr–ZrN–(Zr, Mo, Al)N, obtained by the method of substance condensation by cathode-vacuum-arc deposition with microdroplet phase filtration, for the subsequent determination of the most preferred coating technology.

2. Materials and Methods

2.1. Object of study

The object of this study is multicomponent nanocomposite coatings of the following compositions: Ti–TiN–(Ti, Mo, Al)N; Ti–CrN–(Cr, Mo, Al)N; Ti–Zr–ZrN–(Zr, Mo, Al)N. The substrate material for deposition of these coatings was structural cryogenic steel 12Kh18N10T with high corrosion resistance (Russian Standard 5632–2014 [24]). Table 1 provides information on the thickness, initial components, and elemental composition of multicomponent nanocomposite coatings.

2.2. Coating method

The coatings under consideration were applied using a VIT-2 installation for applying multicomponent nanocomposite coatings. The technological process of applying these coatings on the surface of parts included 4 stages:

– preparatory;

– ion cleaning and heating;

– deposition of multicomponent nanocomposite coatings;

– product cooling.

The preparatory stage is a vacuum pumping in a vacuum system. The starting stage includes cleaning

Table 1. Multicomponent nanocomposite coatings

Coating	Coating thickness, μm	Elemental composition of initial substances	Main characteristics
Ti–TiN–(Ti, Mo, Al)N	4	The TiN system, which contains about 40 at. % Mo and about 10 at. % Al	The nanolayer structure of the studied coatings has a periodic structure, with the value of the nanolayer period λ of 43 nm
Ti–CrN–(Cr, Mo, Al)N		The CrN system, which contains about 40 at. % Mo and about 10 at. % Al	The nanolayer structure of the studied coatings has a periodic structure, with the value of the nanolayer period λ of 48 nm
Ti– Zr–ZrN–(Zr, Mo, Al)N		The ZrN system, which contains about 40 at. % Mo and about 10 at. % Al	The nanolayer structure of the studied coatings has a periodic structure, with the value of the nanolayer period λ of 40 nm

the tool with a glow discharge. Next, ion processing and deposition of coatings on the work piece takes place.

During the study, samples with a multicomponent nanocomposite coating were subjected to hardness, roughness and tribological tests.

2.3. Testing methods

Hardness tests were based on the Russian Standard 8.748–2011 (ISO 14577-1:2002) and taken to measure the hardness (modulus of elasticity during indentation) of experimental samples of multicomponent nanocomposite coatings [25]. To ensure the operability of shut-off valves, the surface hardness (modulus of elasticity during indentation) of samples with multicomponent nanocomposite coatings of different composition was supposed to be approximately 30 GPa.

When considering the quality of coatings, special attention was paid to roughness, which was measured according to the Russian Standard 19300–86 [26].

Tribological tests were carried out to determine the coefficient of dry friction. The essence of the method is as follows: the universal friction machine MTU-1 (TU 4271-001-29034600–2004) implements a test method for friction and wear of materials, which is based on the mutual movement in a horizontal plane of a coated material sample pressed against each other with a given force and counter bodies made of F-4 grade fluoroplast without lubricants.

Table 2 provides information on samples with a multicomponent nanocomposite coating that passed the corresponding test. All tests began with the control of the experimental sample by a visual control method using such measuring instruments as: a magnifying glass according to Russian Standard 25706–83 and a caliper according to Russian Standard 166–89. Samples that did not have visually observable defects were allowed to be tested [27, 28].

Table 2. Test samples

Test type	Sample type	Sample size
Hardness	Plate	50×50×5 mm
Roughness	Plate	50×50×5 mm
Tribology	Cylinder	Diameter 20 mm, length 25 mm

Before testing for hardness, the surface of each test sample was thoroughly cleaned of impurities and degreased. To do this, the surface of the samples was wiped with technical semi-coarse-wool felt according to Russian Standard 6308–71, abundantly moistened in technical hydrolysis rectified ethyl alcohol according to Russian Standard 55878–2013 [29, 30].

2.3.1. Method for determining the modulus of elasticity during indentation

When determining the modulus of elasticity during indentation, measurements were carried out at least in five places on the surface of the test object. The location of the imprint centers was chosen according to the designated control zones (Fig. 1). The test result was calculated as the arithmetic mean of the measurements.

The results of hardness tests were measured after the removal of the test load. Therefore, the effect of elastic deformation of the material under the impact of the indenter tip was not taken into account. By tracing the full cycle of loading and unloading of the test load, it is possible to determine hardness values that are equivalent to those measured by classical hardness measurement methods. Also, this method

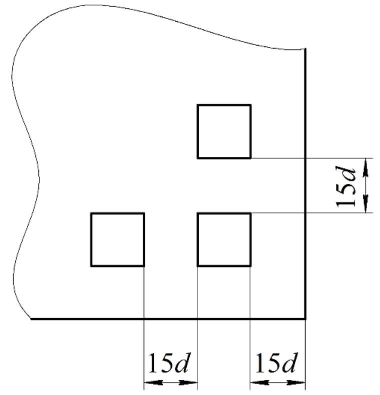

Fig. 1. Control zones on the surface of the test object, on which d is the diameter of the smallest indentation circle

makes it possible to determine additional properties of the material, such as its modulus of indentation elasticity and elastoplastic hardness. Continuous measurement of load values and indentation depth makes it possible to determine the properties of the coating under study, which is schematically shown in Fig. 2.

The values of the test load F and the corresponding indentation depth h were recorded throughout the measurement. As a result, the data were obtained on the applied load and the corresponding indentation depth as a function of time, the F–h-diagram. The value of the indentation elastic modulus E_{IT} was close to the value of the Young's modulus of the material. The value of the E_{IT} modulus was calculated using the formula (1):

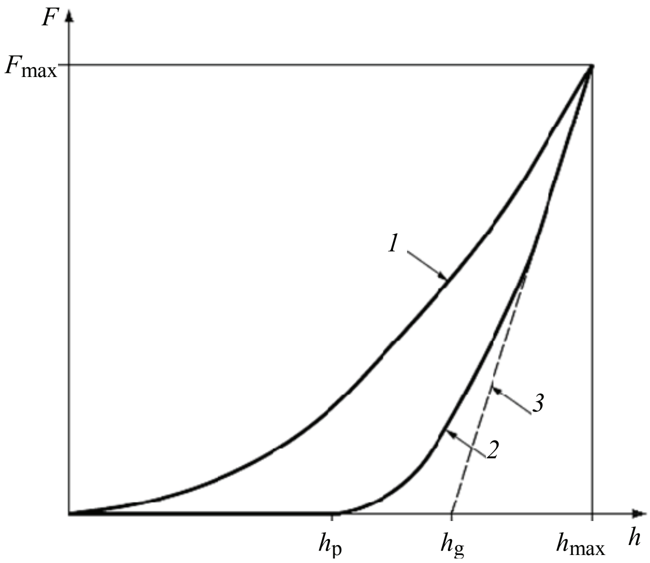

Fig. 2. Correlation between load and indentation depth (F–h-diagram): *1* – curve corresponding to the increase in test load (loading); *2* – curve corresponding to a decrease in the test load (unloading); *3* – tangent to curve *2* for F_{max}

$$E_{IT} = \frac{1-\nu_S^2}{E_R^{-1} - \left(1-\nu_i^2/E_i\right)}, \quad (1)$$

where ν_S is Poisson's ratio of the test material; ν_i is Poisson's ratio of the tip material (for diamond 0.07); E_i is modulus of the tip elasticity (for diamond $1.14 \cdot 10^6$ N·mm^{-1}); $E_R = \sqrt{\pi}\big/2C\sqrt{A_p}$ is reduced modulus of elasticity in the indentation area; $C = \mathrm{d}h / \mathrm{d}F$ is compliance at the point of contact, determined from the load release curve at maximum load (reciprocal of the contact stiffness); A_p is cross-sectional area of the contact surface between the tip and the test sample, determined from the load curve on F–h-diagram and tip area functions.

2.3.2. Method for determining roughness

To carry out tests to determine the roughness, a model 130 profilometer-profilographwas used with the 1st degree of accuracy according to Russian Standard 19300–86 [26]. During testing, the following tracing parameters were set for the profilometer: scanning speed 0.5 mm·s^{-1}; filter λ_b =0.8 mm; scale R_t 50; tracing length 4 mm. The microprofile measurement areas were evenly distributed over the entire surface.

2.3.3. Method for determining the coefficient of dry friction

During tribological tests, samples with a multicomponent nanocomposite coating and an uncoated sample were studied. For the study, counter samples were used, which were uncoated flat disks 50 mm in diameter and 5 mm thick, made of F-4 fluoroplast according to TU 6-05-810–88. After visual control, the test objects were washed with acetone in accordance with Russian Standard 2603–79, after which they were conditioned for at least 88 hours of a standard test atmosphere 23/50 in accordance with Russian Standard 12423–2013 [31, 32].

Tribological tests were carried out using a universal friction machine MTU-1. The distribution of forces and loads in the contact is shown in Fig. 3, where the following designations are accepted: F_{load} is the force acting on the elastic element, F_d is the interaction force of the contacting surfaces of the friction pair, R_c is the average value of the contact spot radius, R_0 is the distance from the rotation circle to the thrust pin.

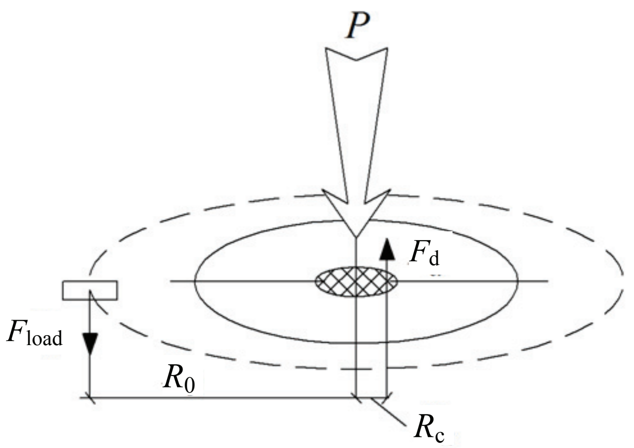

Fig. 3. Distribution of forces and loads in contact

Equating the moment of force in the contact spot and the moment of force of the elastic element, one can calculate the interaction force in the contact F_d using the formula (2):

$$R_0 F_{load} = R_c F_d ;$$

$$F_d = F_{load} R_0 / R_c . \quad (2)$$

Further, the force F_{load} was determined from the calibration curves. The coefficient of dry friction in the tribocouple was calculated by the following formula (3):

$$k_{TP} = \frac{F_d}{P} = \frac{R_0 F_{load}}{R_c P} . \quad (3)$$

3. Results and Discussion

The results of hardness tests are presented in Tables 3–6; graphs of indentation depth versus load for different types of coatings are in shown Figs. 4–6.

When processing the test results, the arithmetic mean values and standard deviations for the values for samples with various types of multicomponent nanocomposite coatings were obtained (see Table 7). From the presented results, one can make an unambiguous conclusion that the surface hardness of coated samples is much higher than for uncoated ones. At the same time, the sample coated with Cr–CrN–(Cr,Mo,Al)N showed the highest value of indentation hardness, while uncoated samples showed the lowest value of indentation hardness.

Figures 7*a–d* show the results of roughness measurement tests; the arithmetic mean deviations of the profile *Ra* and the maximum profile heights R_{max} for samples with different types of coatings are presented in Table 8.

Table 3. Hardness test results for an uncoated metal sample

Name and designation of the test object	Test number	Measured value		
		Indentation hardness H_{1T}, GPa	Indentation elastic modulus E_{1T}, GPa	Elastic recovery η_{IT}, %
Metal sample (steel 12Kh18N10T)	1	2.4	153.7	12.0
	2	2.5	156.8	11.3
	3	2.4	142.1	12.0
	4	2.4	149.5	11.8
	5	2.5	151.4	11.7

Table 4. Results of hardness testing of the Ti–TiN–(Ti, Mo, Al)N coated sample

Name and designation of the test object	Test number	Measured value		
		Indentation hardness H_{1T}, GPa	Indentation elastic modulus E_{1T}, GPa	Elastic recovery η_{IT}, %
Ti–TiN–(Ti, Mo, Al)N coated sample	1	31.7	361.8	61.2
	2	40.4	421.1	67.7
	3	34.2	339.1	66.2
	4	34.79	416.7	62.6
	5	38.8	471.3	59.2

Table 5. Results of hardness testing of the Ti–CrN–(Cr, Mo, Al)N coated sample

Name and designation of the test object	Test number	Measured value		
		Indentation hardness H_{1T}, GPa	Indentation elastic modulus E_{1T}, GPa	Elastic recovery η_{IT}, %
Cr–CrN–(Cr, Mo, Al)N coated sample	1	36.0	349.8	69.3
	2	41.5	359.6	67.3
	3	36.7	345.2	66.8
	4	36.3	340.4	73.3
	5	44.0	413.6	75.3

Table 6. Results of hardness testing of the Ti–Zr–ZrN–(Zr, Mo, Al)N coated sample

Name and designation of the test object	Test number	Measured value		
		Indentation hardness H_{1T}, GPa	Indentation elastic modulus E_{1T}, GPa	Elastic recovery η_{IT}, %
Zr–ZrN–(Zr, Mo, Al)N coated sample	1	23.5	270.6	61.8
	2	25.4	271.9	61.5
	3	23.8	319.5	74.6
	4	28.0	373.6	56.3
	5	24.4	381.7	79.1

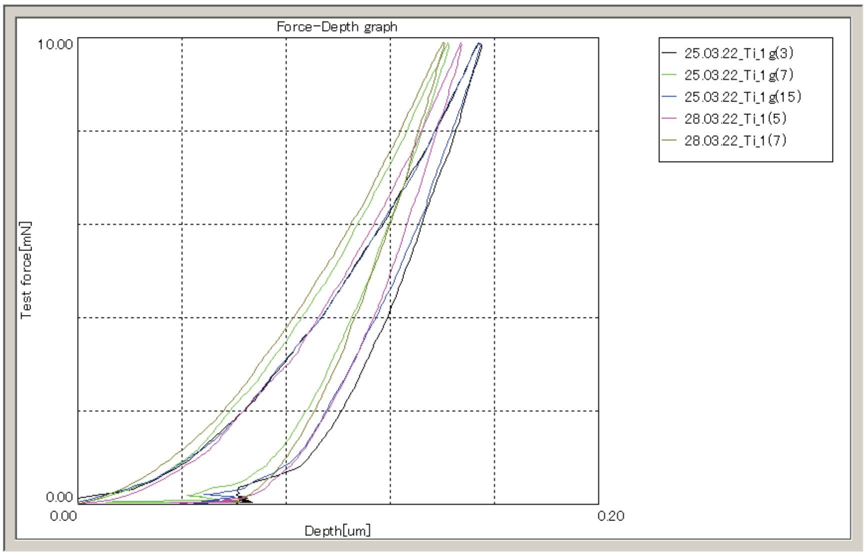

Fig. 4. Graphs of indentation depth versus load for a Ti–TiN–(Ti, Mo, Al)N coated sample

The roughness test results of multicomponent nanocomposite samples showed that Cr–CrN–(Cr, Mo, Al)N had the least roughness of the presented coatings, its arithmetic mean profile deviation R_a was 0.242, and the maximum profile height R_{max} was 0.609.

The tribological test results of samples are shown in Table 9, with σ in Table 8 denoting the standard deviation. The test speed was 5 mm·s^{-1}; the friction path for each specimen was 5 mm; run-in of sample 1 took 16 s; run-in of sample 2 took 65 s; run-in of sample 3 took 78 s; run-in of sample 4 took 52 s.

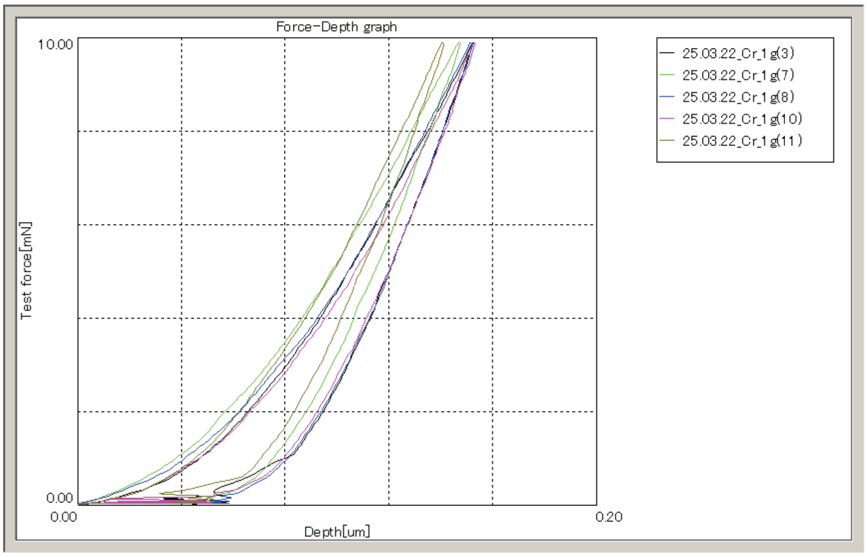

Fig. 5. Graphs of indentation depth versus load for a Cr–CrN–(Cr, Mo, Al)N coated sample

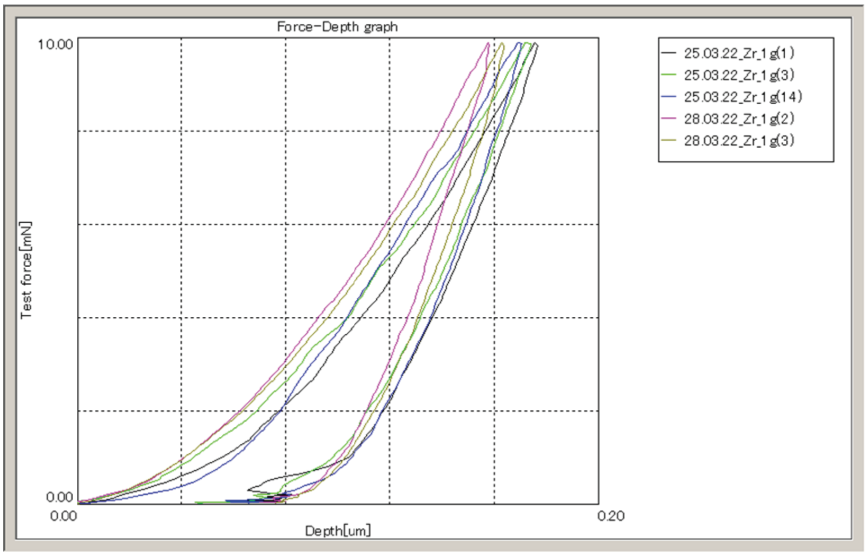

Fig. 6. Graphs of indentation depth versus load for Zr–ZrN–(Zr, Mo, Al)N coated sample

Table 7. Summary of hardness test results

Name and designation of the test object	Design values					
	Arithmetic mean value			Mean square deviation		
	Indentation hardness H_{IT}, GPa	Indentation elastic modulus E_{IT}, GPa	Elastic recovery η_{IT}, %	Indentation hardness H_{IT}, GPa	Indentation elastic modulus E_{IT}, GPa	Elastic recovery η_{IT}, %
Uncoated steel 12Kh18N10T	2.44	150.7	11.8	0.03	5.5	0.3
Ti–TiN–(Ti, Mo, Al)N coated sample	35.9	402.0	63.4	3.5	52.3	3.6
Cr–CrN–(Cr, Mo, Al)N coated sample	38.9	361.7	70.4	3.6	29.9	3.7
Zr–ZrN–(Zr, Mo, Al)N coated sample	25.0	323.5	55.6	1.8	53.3	6.2

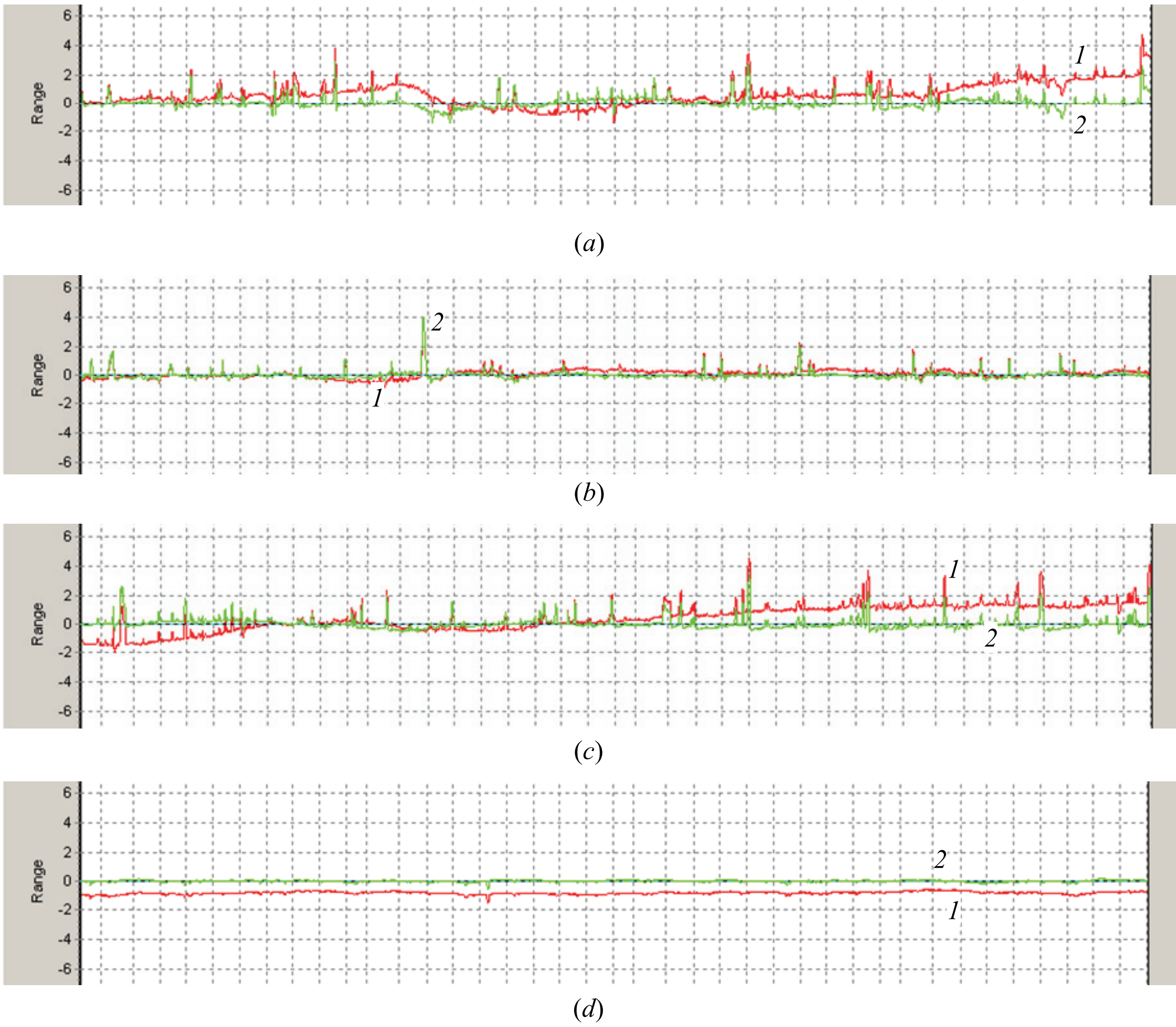

Fig. 7. Roughness profilogram: *a* – Ti–TiN–(Ti, Mo, Al)N coatings; *b* – Cr–CrN–(Cr, Mo, Al)N coatings; *c* – Zr–ZrN–(Zr, Mo, Al)N coatings; *d* – uncoated sample. The profilograms of two independent measurements are shown in red *1* and green *2*

Table 8. Summary of test results

Name and designation of the test object	Measured value	
	Arithmetic mean profile deviation R_a, μm	Maximum profile height R_{max}, μm
Sample coated with Ti–TiN–(Ti, Mo, Al)N	0.362	1.030
Sample coated with Cr–CrN–(Cr, Mo, Al)N	0.242	0.609
Sample coated with Zr–ZrN–(Zr, Mo, Al)N	0.353	0.937

According to the test results, a sample with a Ti–TiN–(Ti,Mo,Al)N coating in a friction pair with a counter-sample from fluoroplast-4 showed the highest coefficient of dry friction, and an uncoated sample in a friction pair with a counter-sample from fluoroplast-4 showed the smallest coefficient of friction. At the same time, among the coated samples, the smallest value of the friction coefficient is for the sample coated with Cr–CrN–(Cr, Mo, Al)N.

Table 9. Roughness test results

Name and designation of the test object	Value		
	Axial force, N	Tangential force, N	Friction coefficient
Ti–TiN–(Ti, Mo, Al)N coated steel disc	155.8 (σ = 0.3996)	18.84 (σ = 1.115)	0.1018 (σ = 0.007145)
Cr–CrN–(Cr, Mo, Al)N coated steel disc	155.8 (σ = 0.4108)	23.81 (σ = 2.308)	0.0806 (σ = 0.01481)
Zr–ZrN–(Zr, Mo, Al)N coated steel disc	155.8 (σ = 0.4109)	20.26 (σ = 1.848)	0.0867 (σ = 0.01186)
Uncoated steel disc	155.8 (σ = 0.4092)	9.08 (σ = 0.2482)	0.0583 (σ = 0.001576)

4. Conclusion

Due to the fact that valves are significant parts of many engineering systems, they must have high reliability and durability. In order to improve these characteristics, new materials, coatings and technologies are being developed and introduced. In this work, the characteristics of multicomponent nanocomposite coatings were studied: Ti–TiN–(Ti, Mo, Al)N; Ti–CrN–(Cr, Mo, Al)N; Ti–Zr–ZrN–(Zr, Mo, Al)N. In the course of testing hardness and roughness, it was found that the best values of the studied characteristics, namely, indentation hardness (38.9 GPa) and roughness (average profile deviation is 0.242 μm) demonstrated samples coated with Cr–CrN–(Cr, Mo, Al)N; according to the results of tribological tests, the lowest coefficient of friction (0.0806) had the Cr–CrN–(Cr, Mo, Al)N coating. Despite these results, further research is needed in this area. A possible promising direction may be the development and creation of new multicomponent nanocomposite coatings based on high-entropy alloys.

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6. Conflict of interests

The authors declare no conflict of interest.

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