DOI: 10.21122/2220-9506-2025-16-2-133-139

Control of Tribological Characteristics of Wear-Resistant AlCrBN Coatings by Nanoscratch Testing

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Received 10.02.2025
Accepted for publication 21.04.2025

Abstract

In recent years, high-precision probe methods have been increasingly used to control the surface microstructure, mechanical and tribological properties of coatings instead of standard methods. The aim of the work was to study the tribological characteristics of the wear-resistant coatings (using the example of AlCrBN coatings deposited with changes in nitrogen pressure, substrate bias voltage and cathode current) at the micro- and nanolevel using the nanoscratch testing (nano-scratching) method. The nanoscratch testing method is a non-standard method of tribotesting the wear-resistant coatings and is based on the reciprocating movement of a spherical diamond indenter with a curvature radius of 226 nm on the surface (under a certain load). It was found that the friction coefficient decreases from 0.087 to 0.036 for coatings deposited with an increase in pressure from 2 to 5 Pa. When the bias voltage on the substrate changes from -50 to -150 V, the friction coefficient decreases from 0.077 to 0.041 and when the cathode current changes from 80 to 100 A, the friction coefficient remains virtually unchanged. The use of this method made it possible to perform multi-cycle tribotesting of the AlCrBN coatings, determine the average values of the friction coefficient, and completely eliminate the influence of microparticles (the characteristic defects for coatings deposited by the cathodic arc method) on the measurements. Thus, the effectiveness of the nanoscratch testing (nano-scratching) as a method for the control wear-resistant coatings is demonstrated.

Keywords: coating, microparticles, roughness, coefficient of friction, nanoscratch testing

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Для цитирования:
Lapitskaya V.A., Kuznetsova T.A., Warcholinski B., Khabarova A.V., Hamzeleva T.V., Gilewicz A., Chizhik S.A.
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Приборы и методы измерений.
2025. Т. 16. № 2. С. 133–139.
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For citation:
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Контроль трибологических характеристик износостойких покрытий AlCrBN методом наноскретчтестирования

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Поступила 10.02.2025
Принята к печати 21.04.2025

В последние годы всё чаще применяют высокоточные зондовые методы для контроля микроструктуры поверхности, механических и трибологических свойств покрытий вместо стандартных методов. Целью работы было исследование трибологических характеристик износостойких покрытий (на примере покрытий AlCrBN, осаждённых при изменении давления азота, напряжения смещения на подложке и тока катода) на микро- и наноуровне с применением метода наноскретчтестирования (наноцарапания). Метод наноскретчтестирования является нестандартным методом трибоиспытаний износостойких покрытий и основан на возвратно-поступательном движении по поверхности (под определённой нагрузкой) сферического алмазного индентора с радиусом закругления 226 нм. Установлено, что коэффициент трения снижается с 0,087 до 0,036 у покрытий, осаждённых при увеличении давлении с 2 до 5 Па. При изменении напряжения смещения на подложке с -50 до -150 V коэффициент трения уменьшается с 0,077 до 0,041, при изменении величины тока катода с 80 до 100 А коэффициент трения практически не меняется. Применение такого метода позволило провести многоцикловое трибоиспытание покрытий AlCrBN, определить средние значения коэффициента трения, а также полностью исключить влияние микрочастиц (характерных дефектов для покрытий, осаждённых катодно-дуговым методом) на измерения. Таким образом показана эффективность наноскретчтестирования (наноцарапания) как метода контроля износостойких покрытий.

Ключевые слова: покрытие, микрочастицы, шероховатость, коэффициент трения, наноскретчтестирование

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Introduction

On the surface of nitride coatings deposited by cathodic arc evaporation method, there is a microdroplet phase (or microparticles ranging in size from the hundreds of nanometers to several micrometers [1–3]. In some works, such structural units are considered coating defects, which can act as concentrators of internal stresses in the coating volume and disrupt its the structural integrity [4]. In other studies, the microparticles can contribute to the formation of a surface modified layer during the friction tests [3]. It should be noted that the attachment of such the microdroplets to the coatings surface is unstable and under the mechanical loads they usually break away from the surface, leaving behind defects in the form of craters and holes. This can also negatively affect the quality and properties of the coatings during operation.

The tribological characteristics of the wear-resistant coatings are usually determined using macro tests on tibomachines using the steel balls with a diameter of the several mm [5–7]. In this case, the tests lead to the deformation and unsuitability of the coating for further use. Such tests participate the microparticles, which can either improve or worsen the tribological properties. To reduce the amount of microparticles on the surface, this is achieved by changing the technological parameters of deposition [8]. Or it is possible to use methods that allow testing on the surface completely eliminating the microparticles. Such methods include the nanoscratch testing method [9–11]. This method eliminates the influence of the microparticles, minimizes the deformation area during tribotesting and allows obtaining results much faster compared to macrotesting due to high contact stresses.

The aim of the work was to demonstrate the efficiency of control the tribological characteristics of the wear-resistant coatings (using the AlCrBN coating as an example) at the micro- and nanolevel using the nanoscratch testing method.

Materials and research methods

The tribological characteristics were tested using nanoscratch testing on the wear-resistant AlCrBN coatings. The AlCrBN coatings with a thickness of 4.4±0.1 μm were deposited using the cathodic arc evaporation method in a TINA 900 M setup [12]. The $Al_{50}Cr_{30}B_{20}$ alloy cathodes were used. The martensitic stainless steel 4H13 (X39Cr13 – DIN standard) was used as a substrate. The substrates 28 mm and 32 mm in diameter and 3 mm thick were polished to a roughness of R_a 0.02 μm. The deposition temperature of AlCrBN coatings 350 °C. A detailed description of the deposition of the coatings is given in [12]. In this work, studies were carried out on coatings obtained with the parameters given in Table 1.

Table 1

Deposition parameters of the AlCrBN coatings

Sample	Nitrogen pressure pN2, Pa	Substrate bias voltage U_B, V	Arc current I_C, A
1	2 Pa	-100 V	80 A
2	5 Pa		
3	4 Pa	-50 V	80 A
4		-150 V	
5	4 Pa	-100 V	80 A
6			100 A

The coating morphology was analyzed using scanning electron microscopy (SEM, LV 5500, JEOL). The Surftest SJ-210 contact profilometer (Mitutoyo, Japan) was used to determine the surface microroughness and the depth of the wear tracks. Three profiles of 2.5 mm long were evaluated on each coating to determine roughness parameters (R_a, R_q, R_z).

The surface morphology and nanoroughness were studied using a Dimension FastScan (Bruker, USA) atomic force microscope (AFM) in the PeakForce QNM (Quantitative Nanoscale Mechanical Mapping) mode. The standard NSC-11 silicon cantilevers (MikroMasch, Estonia) with a cantilever stiffness of 3 N/m and a tip radius of 10 nm were used.

The microtribological properties were determined using a Hysitron 750Ubi (Bruker, USA) nanoindenter with a two-dimensional transducer for nanoscratch testing [11]. A diamond conical indenter with a radius of curvature of 226 nm and an angle of 60° at the apex was used. The load function and the scratches length during testing were set [11]. The tests were carried out in the multi-cycle tests – 100 cycles with a total length of 500 μm and a time of 500 s (1 cycle in 5 s). Load was the 500 μN. The scratch length was 5 μm. The average value of the friction coefficient with standard deviation was determined for 100 cycles. Tribological tests were carried out with reciprocating motion of the indenter.

Results and discussion

According to the SEM study, a large number of microdroplets are present on the surface of the coatings deposited according to the parameters from Table 1 (Figure 1, yellow arrows). Changing the deposition parameters (increasing the pressure from 2 to 5 Pa, the bias voltage on the substrate from -50 to -150 V and the cathode current from 80 to 100 A) leads to a decrease in the number of microdroplets on the surface (Figure 1, *b*, *d*, *f*).

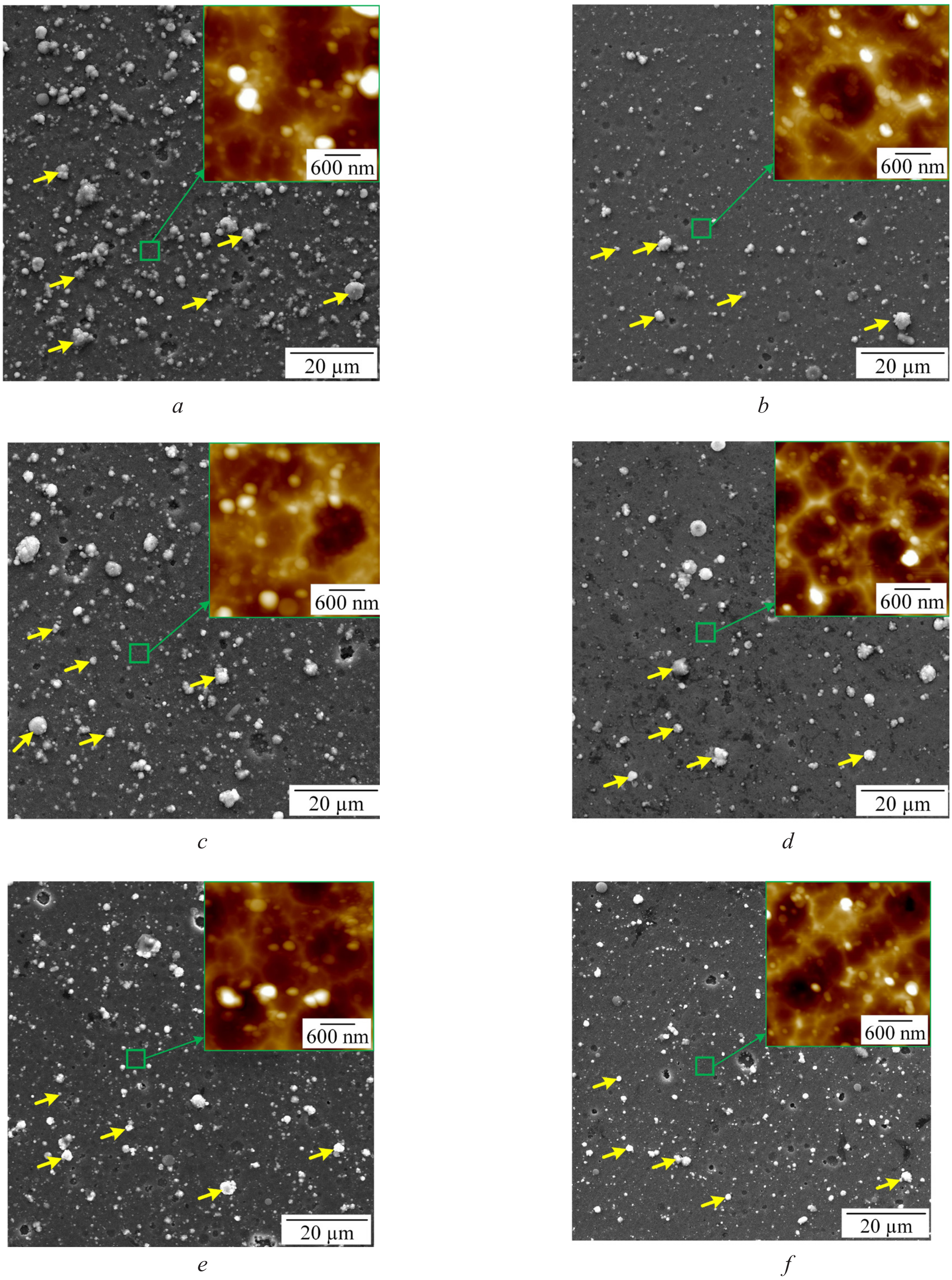

Figure 1 – Scanning electron microscope images (insets – atomic force microscope images, 3×3 μm^2) of the surfaces of the AlCrBN coating applied at different parameters: at 2 Pa, 80 A, -100 V (*a*); at 5 Pa, 80 A, -100 V (*b*); at 4 Pa, 80 A, -50 V (*c*); at 4 Pa, 80 A, -150 V (*d*); at 4 Pa, 80 A, -100 V (*e*); at 4 Pa, 100 A, -100 V (*f*)

For the nanoscratch testing, an area of the several micrometers is sufficient. Therefore, we select the areas on the coating surface within which there are no the microparticles (Figure 1, green squares). The surface structure for testing is shown in the insets (AFM images) in Figure 1.

The presence of microparticles on the surface significantly increases the coatings roughness (Table 2), which in turn affects the tribological characteristics of the coatings. Microroughness measured with an atomic force microscope is significantly reduced in areas without the microparticles (Table 3).

As a result of applying nanoscratch testing on the coatings surface without the microparticles, the dependences of the friction coefficient on the cycles number were obtained (Figure 2). Based on the obtained dependences, the average friction coefficient for the each coating was determined (Table 4).

Table 2

Surface roughness (from a profilometer) of the AlCrBN coatings

Constant parameters	Changing parameters	R_a, μm	R_q, μm	R_z, μm
-100 V, 80 A	2 Pa	0.361 ± 0.044	0.500 ± 0.090	2.496 ± 0.605
	5 Pa	0.236 ± 0.018	0.325 ± 0.039	1.710 ± 0.264
4 Pa, 80 A	-50 V	0.211 ± 0.014	0.275 ± 0.022	1.432 ± 0.147
	-150 V	0.212 ± 0.013	0.281 ± 0.022	1.494 ± 0.150
4 Pa, -100 V	80 A	0.237 ± 0.013	0.323 ± 0.019	1.683 ± 0.074
	100 A	0.189 ± 0.011	0.259 ± 0.019	1.385 ± 0.106

Table 3

Surface microroughness (from an atomic force microscope, on 3×3 μm^2) of the AlCrBN coatings

Constant parameters	Changing parameters	R_a, μm	R_q, μm	R_z, μm
-100 V, 80 A	2 Pa	33.0 ± 1.7	48.9 ± 2.4	219.0 ± 11.0
	5 Pa	21.3 ± 1.1	27.5 ± 1.4	45.4 ± 2.3
4 Pa, 80 A	-50 V	30.3 ± 1.5	41.8 ± 2.1	98.7 ± 4.9
	-150 V	16.9 ± 0.8	21.0 ± 1.1	76.4 ± 3.8
4 Pa, -100 V	80 A	29.0 ± 1.5	38.2 ± 1.9	100.0 ± 5.0
	100 A	14.8 ± 0.7	19.0 ± 1.0	59.8 ± 3.0

Table 4

Tribological properties of AlCrBN coatings by nano-scratch testing and macro testing methods

Constant parameters	Changing parameters	CoF_{micro}	CoF_{macro} [12]
-100 V, 80 A	2 Pa	0.087 ± 0.006	0.67 ± 0.02
	5 Pa	0.036 ± 0.004	0.66 ± 0.02
4 Pa, 80 A	-50 V	0.077 ± 0.018	0.65 ± 0.02
	-150 V	0.041 ± 0.003	0.77 ± 0.01
4 Pa, -100 V	80 A	0.047 ± 0.007	0.70 ± 0.01
	100 A	0.045 ± 0.003	0.72 ± 0.02

The friction coefficient values are an order of magnitude lower than after macrotests (Table 4). The correlation of the friction coefficient after the nanoscratch testing with macrotest is present only for coatings deposited at different pressures – the friction coefficient decreases from 0.087 to 0.036. When the substrate bias voltage changes from -50 to -150 V, the friction coefficient decreases from 0.077 to 0.041. The friction coefficient remains virtually unchanged for coatings when the cathode current changes. Such friction coefficient values are primarily related to the contact area during testing [11]. In macrotests, the microparticles participate in friction in addition to the main coating.

Also, to determine the average friction coefficient, the values for the first 10 cycles were not taken into account, since these cycles are significantly affected by the surface topography (even without microdroplets on the surface, there is a certain surface unevenness). The AFM images (Figure 1) show a cellular structure, which forms the microrelief of the surface on which the nanoscratch testing was carried out.

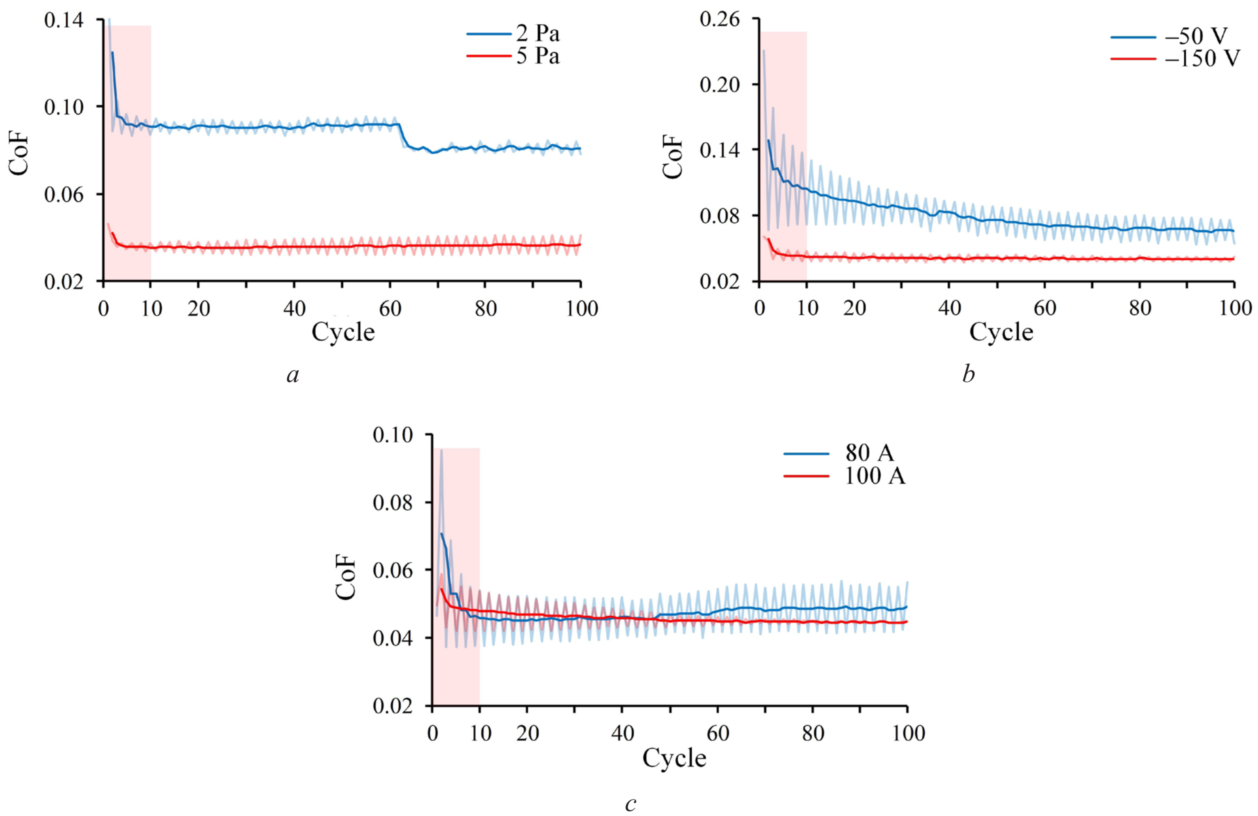

Figure 2 – Dependences of the friction coefficient on the cycles number for AlCrBN coatings applied at different parameters: at 2 and 5 Pa (*a*); at -50 and -150 V (*b*); at 80 and 100 A (*c*)

The zigzag curve on each plot in Figure 2 shows the reciprocating test pattern – the forward and reverse stroke of the diamond spherical indenter.

Conclusion

The tribological characteristics of the wear-resistant AlCrBN coatings deposited by the cathodic arc evaporation method have been studied. The coatings were deposited with changing nitrogen pressure, substrate bias voltage, and cathode current. Tribological tests were carried out at the micro- and nanolevel using the nanoscratch testing method. It was found that the friction coefficient decreases from 0.087 to 0.036 for coatings deposited with an increase in pressure from 2 to 5 Pa. When the substrate bias voltage changes from -50 to -150 V, the friction coefficient decreases from 0.077 to 0.041, and when the cathode current changes from 80 to 100 A, the friction coefficient remains virtually unchanged. The use of the nanoscratch testing method made it possible to exclude the influence of microparticles on the measurements of tribological characteristics. A comparison of the friction coefficient determined by the macrotribotest and nanoscratch testing was carried out. The efficiency of the nanoscratch testing as a method for control the wear-resistant coatings is demonstrated.

Acknowledgments

This research was supported by the grant of Belarusian Republican Foundation for Fundamental Research BRFFR No. T21MS-029. Publication partly financed by the National Centre for Research and Development, Poland, BIOSTRATEG3/344303/14/NCBR/2018.

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