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THE INFLUENCE OF MICROSTRUCTURE OF Cr/CrN NANOMULTILAYER COATINGS ON THEIR TRIBOLOGICAL AND MECHANICAL PROPERTIES

Key words: multilayer coatings, PVD, tribological tests.

Abstract: One of the most effective ways of shaping the operating parameters of functional elements is the modification of the surface layer by modern surface engineering technologies. The most perspective directions in the development of surface engineering solutions are multilayer coatings. The appropriate selection of parameters of multi-layered structure such as thickness, chemical composition, microstructure, and the number of component layer in these type coatings enable effective shaping of their properties. However, a characteristic of the mechanical and tribological properties as a function of parameters of a multilayer structure are still unclear in literature.

The paper presents the influence of multilayer structure on mechanical and tribological properties of the coatings prepared by arc vacuum technics. The analysis of mechanical properties included hardness, Young's modulus, and adhesion tests. The analysis of microstructure was performed using Scanning Transmission Electron Microscopy (STEM). The tribological properties were examined using the pin-on-disc produced by the DUCOM Company. The authors indicated that the multilayer coatings Cr/CrN were characterized by good mechanical and tribological properties. In this paper, the authors confirmed that parameters of the process of the multilayer creation are important in shaping the unique tribological properties of the multilayer coatings.

Wpływ mikrostruktury na właściwości mechaniczne oraz trybologiczne powłok nanowielowarstowych Cr/CrN

Słowa kluczowe: powłoki wielowarstwowe, PVD, badania tribologiczne.

Streszczenie: Jednym z najbardziej efektywnych sposobów kształtowania właściwości eksploatacyjnych elementów użytkowych jest modyfikowanie warstwy wierzchniej poprzez zastosowanie nowoczesnych technologii inżynierii powierzchni. Głównym kierunkiem rozwoju rozwiązań inżynierii powierzchni stały się w ostatnich latach między innymi powłoki wielowarstwowe. Stwarzają one bardzo duże możliwość w zakresie kształtowania ich właściwości poprzez odpowiedni dobór struktury wielowarstwowej, w tym: grubość, ilość, mikrostruktura oraz skład chemiczny warstw składowych. Nadal jednak charakterystyka ich właściwości mechanicznych oraz trybologicznych w funkcji parametrów ich wielowarstwowej konfiguracji pozostaje niejasna w dostępnej literaturze.

W artykule przedstawiono wpływ parametrów budowy wielowarstwowej powłok Cr/CrN wytwarzanych metodą łukowo-próżniową na ich właściwości mechaniczne oraz tribologiczne. Analiza właściwości mechanicznych obejmowała badania twardości, modułu Younga oraz badania adhezji metodą zarysowania. Analizę mikrostruktury przeprowadzono z wykorzystaniem skaningowej mikroskopii transmisyjnej (STEM). Badania właściwości tribologicznych zostały przeprowadzone w temperaturze pokojowej z wykorzystaniem testera tribologicznego firmy DUCOM typu pin-on-disc. Autorzy wskazują, iż powłoki nanowielowarstowe Cr/CrN charakteryzują się dobrymi właściwościami mechanicznymi oraz dobrymi właściwościami tribologicznymi. W artykule autorzy potwierdzili wpływ parametrów budowy wielowarstwowej powłok PVD w procesie kształtowania ich właściwości trybologicznych.

Introduction

One of the most effective ways of shaping the operating parameters of functional elements is to modify the surface layer using modern technologies of surface engineering. In recent years, the main directions in the development of surface engineering solutions are multilayer coatings, which enable effective shaping of surface layer properties [1–3]. One example of a multilayer coating is Cr/CrN coating, whose properties are very well described (reported) in the literature [4–6]. Coatings are characterized by excellent mechanical and tribological properties due to the presence of hard layers based on nitrides and a plastic metallic layer in the structure. In this connection, the plastic layer of metallic Cr can absorb excessive plastic deformation, while the phase of hard chromium nitride ensures good wear resistance. In addition, the presence of boundaries between the component layers in the Cr/CrN coating structure ensures high brittle cracking resistance. The presence of transition layers could change the cracking mechanism of the coating from a single-step, which is characteristic for single-layer coatings, to a multi-stage/steps mechanism. The initiation of cracks in the coating can come from both the substrate and from the surface. The propagation of cracks in the single-layer coating can destroy it in the whole cross-section. However, in the multi-layer coating, the boundaries between the component layers are places of energy dissipation. The directions of microcracks propagation can change, thus preventing their further spreading in the coating. This phenomenon prolongs the path of a single crack. At the same time, the energy is reduced and significantly improves the resistance of multi-layer coatings to brittle fracture [7–8].

Thanks to these properties, the Cr/CrN multilayer coating can be successfully used to improve durability on plastic deformation and erosion processes, which was confirmed in the many research works [9–10]. The large possibilities of using Cr/CrN multilayer coating are connected with the possibility of practically optional designing of the surface layer properties by shaping of parameters of their multilayer structure (phase composition, chemical composition, microstructure, grain size, and state of stress) and the whole coating (number and thickness of component layers).The literature analysis confirmed the possibility of shaping the mechanical properties of the Cr/CrN coating by changing the multi-layer configuration [11]. It should be noted that the characteristic of tribological properties as a function of the parameters of the multilayer configuration of Cr/CrN coatings is still unclear in the available literature.

The authors of article present the analysis of the properties of Cr/CrN coatings that are produced by the arc vacuum method depending on the number of component layer in the range of 10–100. An analysis of the impact of the multilayer configuration on their tribological properties is also presented. The research methods which were presented in the article include the analysis of mechanical properties and microstructure and the analysis of tribological properties of selected nano-multilayer coating in the function of the number and thickness of component layers.

1. Experimental details

1.1 Deposition of coatings

Samples for investigations were made of titanium alloy Ti6Al4V. Before the deposition process, substrates were cleaned and mechanically polished using the procedure reported in literature [12].

The investigated PVD multilayers coating were created by means of the Arc Evaporation method, with the use of CDS-Standard device designed and produced by the Łukasiewicz Research Network-Institute for Sustainable Technologies, Radom. The technological processes include the formation of three coatings of different thicknesses and the number of component layers. All coatings were made using four pure Cr targets. Parameters of the surface treatment technology are presented in Table 1.

Table 1. Parameters of the deposited PVD coatings

Coating	Layer	Atmosphere	Temperature T [°C]	Pressure p [mbar]	Voltage U_{bias} [V]	Current I_{bias} [A]	Time t [min]
Cr/CrN 10	Cr	100%Ar	350	5.0×10^{-3}	- 50	4x50	15
	CrN	100%N_2		3.5×10^{-2}	- 200	4x50	7
Cr/CrN 50	Cr	100%Ar	360	5.0×10^{-3}	- 50	4x50	3
	CrN	100%N_2		3.5×10^{-2}	- 200	4x50	1.4
Cr/CrN 100	Cr	100%Ar	340	5.0×10^{-3}	- 50	4x50	1.5
	CrN	100%N_2		3.5×10^{-2}	- 200	4x50	0.7

1.2. Characteristics of the mechanical properties of coatings

1.2.1. Hardness and Young's modulus test

Mechanical properties like hardness and Young's modulus for investigated coatings were measured by means of the nanoindentation method using a Nano-Hardness Tester by CSM Instruments with a Berkovich diamond indenter. The hardness and Young's modulus measurements of selected PVD coatings were carried out according to parameters presented in Table 2. The maximum penetration of the indenter is $h_{max}<0.1$ (10%) of the thickness of the layer. Parameters used in measurements of Young's modulus using the Nano-Hardness Tester are given in Table 2.

Table 2. Results of measurements of hardness and Young's modulus by means of the Nano-Hardness

Coating	Maximum penetration of indenter h_{max} [nm]	Maximum load of indenter F_{max} [mN]	Number of load cycles	Dwell time of maximum load [s]	Type of indenter
Cr/CrN 10	400	61	1	1	Berkovich
Cr/CrN 50	400	62	1	1	Berkovich
Cr/CrN 100	400	63	1	1	Berkovich

The load and displacement of intender were recorded during all measurements. The next step was the plotting of the displacement-dependence curve as a function of the load.

In accordance with the mathematical principles developed by Oliver and Pharr, both of values of the hardness of the tested coatings and the Young's modulus were determined [13]. Based on the results of the hardness and Young's modulus, the "plasticity index" H^3/E^2 was determined, which is widely quoted as a valuable measure in determining the limit of elastic behaviour in a surface contact, and it was clearly important for the avoidance to wear [14].

1.2.2. Adhesion test

Adhesions were measured using a scratch-test method with the use of a Revetest (CSM Instruments) equipped with a diamond Rockwell indenter with a radius of 200 μm and an apex angle of 120º. The indenter's load force increased linearly in the range of 0-200N, and the loading rate was 10 N/min. The scratches were 5 mm. During the test, the changes of acoustic emission (AE) and friction forces (Ft) as a function of the force loading the indenter (Fn) were registered. Each sample was subjected to three such tests. The scratch specimens were also subjected to a detailed examination by SEM after the test series had been completed. Based on the changes in acoustic emission and friction force, three critical loads were determined corresponding to completely damage of the coating. The Fc1 parameter described the value of the load on the penetrator under which the first fractures were observed, the Fc2 parameter described the load under which the next adhesive damages in the form of spalling occurred, and the Fc3 parameter was related to the load under which decohesion occurred in the entire layer from the substrate.

1.3. Surface and microstructure characterization

The microstructures of coatings were characterized with Scanning Transmission Electron Microscopy (STEM) Hitachi HD2700. The measurements were investigated on samples which were made of titanium alloy Ti6Al4V. For the preparation of samples, a Hitachi FB2100 was used with a focused ion beam (FIB) system equipped with a Ga+ion source with accelerating voltage in the range 10–40 kV and the current in the range 0.01–45 nA. The thickness of STEM thin foil was about 100 nm.

1.4. Tribological properties of coatings

Tribological tests of samples were executed using the pin-on-disc tribometer produced by the DUCOM Company. The investigated samples were prepared from the Ti6Al4V alloy. For all of our samples, the tests were performed at room temperatures for the combination with Al_2O_3 ball–shield covered with the test coating in accordance with the parameters shown in Table 3.

Table 3. Parameters of pin-on-disc tribological tests

Temperature [°C]	Ball load force [N]	Wear track [m]	Number of cycles	Sliding distance [m/s]	The diameter of wear track[mm]	The radius of wear track [mm]
25	2	350	6370	0.2	15	6

After the tests, wear tracks were scanned using an Interferometric Profilometer, and the volume of the removed coating material was measured. Based on the volume of removed coatings material, the wear index W was calculated, according to the following formula:

$$W = V / F \cdot s,$$

where

V – volume of removed coatings material [mm^3],
F – ball load force [N],
s – length of wear track [m].

To determine the dominant mechanism of the destruction during the scratch test, the created wear tracks were also subjected to microscopic observation and measurements of chemical composition by using the EDS technique.

2. Results and discussion

2.1. Microstructure and chemical composition

The results of the microstructure and cross section analysis are presented in Fig.1. The surface analysis of coatings revealed a small variation of the surface roughness. As the number of component layers increased, the significant roughness was changed.

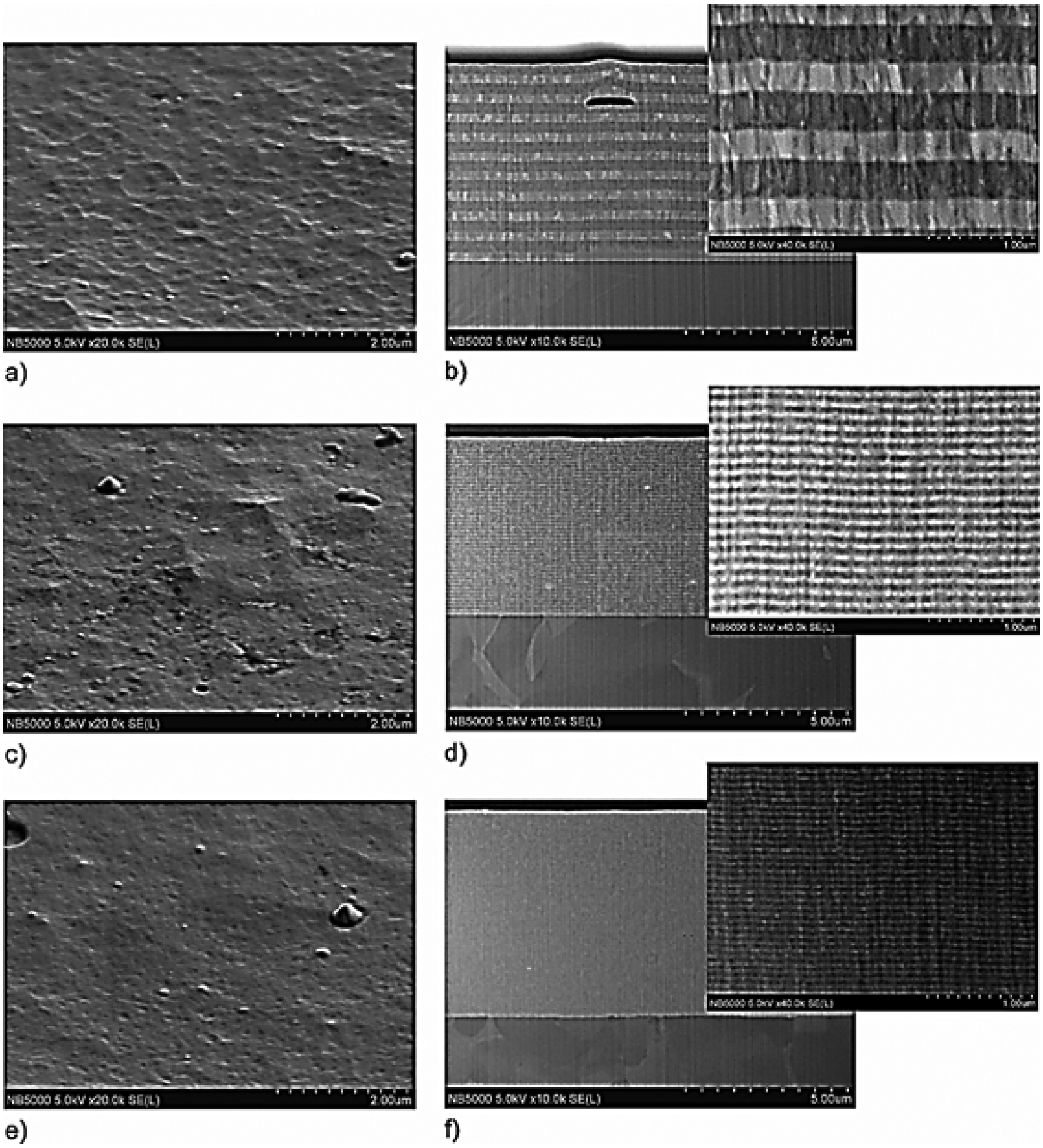

Fig. 1. Microstructure of investigated coatings: a) Cr/CrNx10 coating-surface, b) Cr/CrNx10 coating–cross-section, c) Cr/CrNx50 coating-surface, b) Cr/CrNx50 coating-cross-section, e) Cr/CrNx100 coating-surface, f) Cr/CrNx100 coating-cross-section

The conducted analysis has also confirmed a significant differentiation in the thickness for the component layers in the obtained coatings. The parameters of the multilayer coating are presented in Table 4. In all cases, the total thicknesses of the deposited coatings were similar and were in the range of 5.2–5.8 μm. It was observed that coatings also have a homogeneous thickness for all component layers. The increasing number of the component layers can affect the reduction in the thickness of individual of Cr/CrN layers. The ratio for the thickness of metallic Cr layer and CrN layer is at the level of about 1:1. In the case of the coating which consists of 10 Cr/CrN layers, the thickness of the individual layer was about $g_{Cr/CrNx10}$=0.57 μm. In the case of coatings that consisted of 50 and 100 layers, the thicknesses of individually layer were significantly smaller and were respectively p: $g_{Cr/CrNx50}$=0.08 μm, $g_{Cr/CrNx100}$=0.05 μm.

Table 4. Parameters of multilayers coatings

	Thickness			Bi-layer period [μm]	width of columnar grains [μm]
	Total thickness [μm]	Cr [μm]	CrN [μm]		
Cr/CrN 10	5.8	0.24	0.33	0.57	0.09
Cr/CrN 50	5.8	0.03	0.05	0.08	0.05
Cr/CrN 100	5.2	0.02	0.03	0.05	0.04

The detailed observation of multilayer microstructure has indicated that all investigated coatings were characterized by non-porous microstructure with fine dense fibrillar crystallites. This microstructure is typical for the structure of the transition zone T according to the Thorton model proposed for PVD coatings [15]. The increasing of the component layers contributed to a significant fragmentation of the structure. The highest reduction of grain size reduction was observed when the number of Cr/CrN complexes increased from 10 to 50. For the Cr/CrNx10 coating, the size of the columnar grains was about 0.09 μm. For Cr/CrNx50 coating, the size of the grains was about 0.05 μm. Increasing the number of layers did not influence the clear fragmentation of the structure. The grain size for the Cr/CrNx100 coating was decreasing to a value of about 0.04 μm.

2.2. Mechanical properties

The results of the investigations of mechanical properties for coatings are presented in Table 4. The measurements of the hardness and the Young's modulus results showed that increasing number of component layers to 50 causes a significant increase in the value of hardness and Young's modulus. Further increasing of the number of component layers contributed to the decrease of Young's modulus and the lack of changes in hardness. The Young's modulus decreases and changes the elastic properties defined by the H/E plasticity index, which is an important criterion of resistance against plastic deformation of hard coatings. The high H/E value at high hardness indicates very good wear resistance for thin coatings [16]. The analysis of the elastic properties demonstrated that, with the increase in the number of layers, the susceptibility of deformation is reduced, i.e. for all coatings, $H/E_{Cr/CrNx10}$=0.7 and $H/E_{Cr/CrNx50}$=0.8, and, for the coating which consists of 100 layers, $H/E_{Cr/CrNx100}$=0.9. With an increasing thickness of component layers, the resistance to the plastic deformation, which is defined the H^3/E^2index, increases (Table 5). Coatings with high hardness and a small modulus of elasticity will be able to transfer much higher loads, and they not induce plastification [17].

Table 5. Mechanical properties of deposited layers

Coating PVD	Hardness [GPa]	The Young's modulus [GPa]	H/E	H3/E2
Cr/CrN 10	20±2	291±20	≈0.07	≈0.09
Cr/CrN 50	26±2	311±20	≈0.08	≈0.18
Cr/CrN 100	26±1	297±11	≈0.09	≈0.20

The adhesion tests were carried out making three scratches for each of the tested coatings, and then the average values of critical loads were determined. The obtained results showed that the best adhesion to the substrate was characterized by (Cr, CrN)x10 coating. The complete removal of the coating from the substrate was observed at $Fc3_{Cr/CrNx10}$ = 80 N. In the scratch test, the first cracks for this coating were observed with a critical load $Fc1_{Cr/CrN10}$ = 9 N. These cracks were curved opposite to the direction of the indenter movement and propagated outside the area of scratches, and increasing the load generated their multiplication.

For $Fc2_{Cr/CrN10} = 59$ N, it was observed that the coating was only removed on the edges of scratches. Chips were not observed over the entire length of the scratches.

For other coatings, the load values when the total coating was removed from the crack area were significantly lower and were, respectively, $Fc3_{Cr/CrNx50} = 75$ N and $Fc3_{Cr/CrNx100} = 69$ N. The first cracks for these coatings also appeared at a lower load than in the case of the coating which consists of 10 layers ($Fc1_{Cr/CrNx50} = 6$ N and $Fc1_{Cr/CrNx100} = 5$ N). These cracks have only appeared on the edges of the scratches and were arranged parallel to the indenter movement. The appearance of the first adhesive defects in the form of chips were observed when further increasing the load. These chips were observed at $Fc2_{Cr/CrN50} = 24$ N and $Fc2_{Cr/CrN100} = 26$ N. The surfaces of chips were larger for the coating which consists of 100 component layers than for the coating which consists of 50 component layers.

2.3. Tribological properties

The results of the tribological tests at the room temperature are shown in Table 6. In accordance with the calculated the wear index, the best resistance was characterized by the coatings with 100 component layers ($Wz_{Cr/CrN} = 1.08^{-3}$ mm^3/N*km). The coating which consists 50 component layers were characterized by a lower wear index ($Wz_{Cr/CrN50} = 5.93\ ^{e-3}$ mm^3/N*km). The worst resistance for tribological tests were recorded for the coating which consisted of 10 component layers (Cr/CrN 10). The wear index for Cr/CrN50 ($Wz_{Cr/CrN50} = 5.47\ ^{e2}$ mm^3/N*km) was lower. The results of research showed that the wear resistance of the Cr/CrN multilayer coatings depends on the thickness of the Cr and CrN layers. The wear indexes of component layers are decreasing with increasing the thickness of coatings.

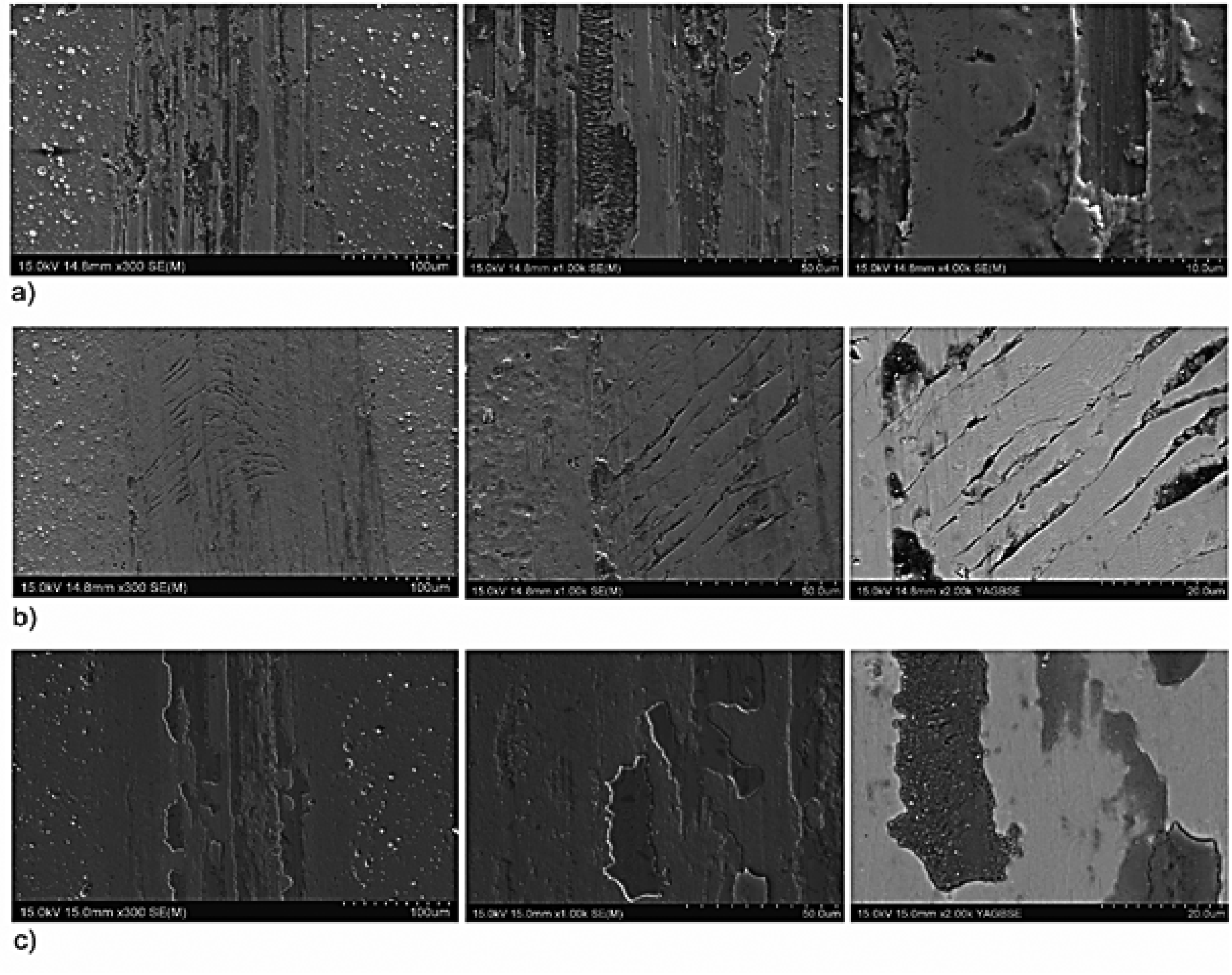

Fig. 2. Images of wear tracks after tribological tests at room temperature: a) Cr/CrNx10 (300x,1000x,4000x), b) Cr/CrNx50 (300x, 1000x, 2000x), c) Cr/CrNx100 (300x, 1000x, 2000x)

Table 6. The wear track index based on the tribological tests

Coating	Volume of wear track [mm^3]			Average volume of wear track [mm^3]	Wear index Wz [mm^3/N*km]
Cr/CrN 10	5.98E-03	9.93E-02	9.58E-03	3.83E-02	5.47E-02
Cr/CrN 50	9.57E-03	1.64E-03	1.24E-03	4.15E-03	5.93E-03
Cr/CrN 100	6.95E-04	8.60E-04	7.07E-04	7.54E-04	1.08E-04

The analysis of wear tracks after tribological tests (Fig. 2) showed that different destruction mechanisms of the coating were observed. The results of the tribological tests depended on the number of multilayers parameters. The area of the wear for 10 layers (Cr/CrN10) was characterized by non-homogenous surface (Fig. 2a). We can observe a lot of grooves and indentations which were characteristic of the abrasive wear mechanism through grooving. In the results of this phenomenon, cooperation at the interfaces of layers was worse, and this leads to faster damage of the coating. The abrasive wear of this coating was increased by surface fatigue. It led to separating relatively large fragments which were crushed, and they were present at friction contact. The analysis of the chemical composition did not show the presence of aluminium, which indicates a lack of wear products of the Al_2O_3 counter-sample.

The analysis of wear track for other samples (Cr/CrN50 and Cr/CrN100) showed a different mechanism of the destruction of these materials in friction processes. The wear destructions, small cracks, and chips were dominant in the scratch which was formed on Cr/CrN50 coating (Fig. 2b). Abrasive and adhesive wear was also observed. We can observe that aluminium was present in wear tracks. It can be information for us about abrasion and the transferring of material from the counter-sample. The larger adhesive destructions in the form of chips were noticed for the Cr/CrN100 coating in the abrasion track (Fig. 2c). The chemical analysis showed that the wear material from counter-body was absent. We can observe the high content of oxygen and chromium on the wear surface. It indicates the oxidation of the coating. It was reported in literature that the Cr_xO_y [18] was formed on the surface of Cr at room temperatures. The presence of Cr_xO_y in a friction pair can reduce the wear [19]. We can observed a typical abrasive wear of the interlayer in the form of a chromium oxide lubricating film (CrxOy), which protected surface coatings against wear [20–21].

3. Conclusion

The analysis of the obtained results confirmed the significant influence of the parameter of the multilayer configuration of coatings in the formation of tribological and mechanical properties. The increasing of hardness was observed with the increasing number of component layer from 10 to 50. This is due the fact that the increasing of the thickness Cr/CrN layer can reduce the thickness of the plastic chromium phase Cr characterized by the low hardness (H_{Cr}=4GPa). The observed decrease in the thickness of the chromium metal layer also caused an increase in the resistance to plastic deformation ($H/E_{Cr/CrN10}$=0.7, $H/E_{Cr/CrN10}$=0.8, $H/E_{Cr/CrN10}$=0.9) for whole complex. This was also confirmed by the results of adhesion tests. For the coating which can be characterized by a low H/E index, chips were not observed over the entire length of the crack. There was only the loosening of the coating from the substrate at the edges of the crack mainly due to the uplift of the material, which is characteristic for materials with high susceptibility to plastic deformation. The critical normal force tends to increase with increasing coating hardness. In the case of other coatings, the appearance of many chips characteristic for brittle materials was observed. Increasing the number of Cr/CrN layers contributed to increasing the wear tracks.

The authors also confirmed the significant effect of the parameter of multilayer configuration of the coatings on tribological properties. The analysis of the results indicated that, with the increasing number of component layers, the abrasion resistance of the tested coatings increase. The change in the thickness of the component layers also affects the change of destructive mechanisms occurring in the process of tribological wear from typical wear for soft materials through grooving on abrasive and adhesive wear. Important is the fact that, with increasing the number of the component layers to 100, Cr/CrN complexes caused an increase in the number of defects in the form of shallow adhesive chips. It caused that the layer of metallic chromium was exposed and was oxidized. The phenomenon of the formation of the Cr_xO_y oxide layer in accordance with the literature has a positive effect on the reduction of wear intensity in tribological friction processes.

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