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OPTIMISING TRIBOLOGICAL PROPERTIES OF ANTI-WEAR AlCrN COATINGS DEPOSITED BY THE CATHODIC ARC METHOD

OPTYMALIZACJA WŁAŚCIWOŚCI TRIBOLOGICZNYCH POWŁOK AlCrN OSADZANYCH METODĄ ŁUKOWĄ

Key words:

AlCrN coatings, cathodic arc deposition, coating anti-wear properties, Taguchi method

Słowa kluczowe:

powłoki AlCrN, osadzanie metodą łukową, właściwości przeciwzużyciowe, metoda Taguchi

Abstract

The Taguchi method was applied to achieve the optimum anti-wear performance of AlCrN hard coatings deposited by the cathodic arc evaporation technique. Four deposition processes parameters i.e. nitrogen pressure, arc current, bias voltage and substrate temperature with three levels were selected

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to estimate the influence of each factor on the coatings defects and their mechanical and tribological properties. Based on the statistical analyses, the optimum deposition conditions were determined to manufacture AlCrN coatings with the best tribological behaviour. The results demonstrate that, the nitrogen pressure, arc current and the bias voltage influence the volume wear of the coating to the greatest extent. The results of the study of the surface topography and the mechanical properties of the AlCrN coatings showed that, in order to obtain the greatest resistance to wear, the coating must be sufficiently smooth (with a low number of surface defects), hard and demonstrate a good adhesion to the substrate. The optimum deposition conditions of the AlCrN coatings with the greatest resistance to wear are as follows: the nitrogen pressure 2.6 Pa, arc current 50 A, negative bias voltage 150 V and substrate temperature 465°C.

INTRODUCTION

PVD hard coatings with good anti-wear properties are nowadays largely used in industrial practice **[L. 1]** Among numerous types of PVD coatings a ternary Al-Cr-N hard coating is being increasingly used for improvement of durability and reliability of cutting **[L. 7–8]** or punching **[L. 9]** of metals and their alloys as well as for wood machining **[L. 10]** due to excellent physical and chemical properties of the coating as, e.g., high hardness (22–34 GPa) **[L. 2]**, high oxidation resistance up to 900°C) **[L. 3–5]** and superior wear resistance **[L. 6]**. The reactive cathodic arc evaporation technique compared with other deposition methods is a very effective technology for industrial manufacturing of AlCrN coatings **[L. 11–12]**. The method renders easy modification of mechanical and tribological properties of the coatings by changing basic deposition parameters as: reactive gas flow rate or pressure, arc current intensity, negative bias voltage or substrate temperature **[L. 6, 13]**. The conventional single-variable design of experiment method (DOE) is very popular to state the effect of each parameter of PVD process on coating properties **[L. 6]** but many experiments must be done to obtain detailed information about influence of the process parameters on the coating quality. The use of the Taguchi technique **[L. 14–15]** based on orthogonal array table (OA) allows reducing the number of experiments and helps to decrease costs and time of research. In this study the influence of selected deposition process parameters on the wear resistance of AlCrN coating was evaluated with use of the Taguchi method. Moreover, the optimum deposition conditions for manufacturing of AlCrN coatings with of the highest wear resistance were determined.

EXPERIMENTAL PROCEDURE

The CrAlN coatings were deposited by reactive cathodic-arc method onto 6 mm thick 25,4 mm diameter discs from 1.2365 hot working steel quenched and tempered to 50±2 HRC. Before deposition the steel substrates were mirror polished (to the surface roughnes R_a = 0.02 μm) and cleaned in in multi-stage ultrasonic bath with trichloroethylene. After mounting in the vacuum chamber the substrates were pre-heated by using I.R. heaters at a residual pressure of 5.0×10^{-5} mbar and temperature of 200°C. Next the substrates were etched in a two-stage process. In the first step Ar^+ ions were used for substrates' surface sputtering at a pressure of 5.0×10^{-3} mbar and at negative substrate bias voltage of 50–300 V. In the second step the etching of the substrate surface was executed with use of Ti^+ ions at a residual pressure less then $5x10^{-3}$ Pa and at a negative substrate bias voltage of -950 V. The AlCrN coatings were deposited with use of five $Al_{0.7}Cr_{0.3}$ composite metal cathodes at reactive nitrogen atmosphere. Four deposition processes parameters, i.e. nitrogen pressure, arc current, bias voltage and substrate temperature with three different levels were selected to estimate the influence of each factor on the coatings defects and the mechanical and tribological properties of the coatings (**Tab. 1**).

Table 1. Parameters and levels selected for the deposition processes of AlCrN coatings
Tabela 1. Wytypowane do badań parametry procesu osadzania powłok AlCrN i ich wartości na poszczególnych poziomach zmienności

Parameters		Levels		
		1	2	3
Nitrogen pressure (Pa)	A	0.8	1.7	2.6
Arc current (A)	B	50	55	65
Bias voltage (V)	C	50	75	150
Substrate temperature (°C)	D	400	430	465

The Taguchi orthogonal array L_9 consisting of nine individual experiments was performed to establish the plan of experiments for establishing the optimum parameters during deposition of the AlCrN coatings (**Table 2**). The commercial vacuum equipment MZ-383 Metaplas Ionon was used for coatings' deposition. In order to achieve better adhesion of the AlCrN coating to the substrate at first a thin 0.1 μm intermediate AlCr layer was deposited onto the steel substrates.

The topography of the coatings' surface was examined with use of an optical profilometer Talysurf CCI Taylor-Hobson and scanning electron microscope (Hitachi S-2460N) and the coatings' roughness with use of a profilometer HOMMEL TESTER T1000. The hardness and elastic modulus of the coatings were measured with a CSM Nano-Hardness Tester with use

Table 2. Deposition conditions for AlCrN coatings based on the Taguchi orthogonal array L_9
Tabela 2. Parametry osadzania powłok AlCrN opracowane w postaci ortogonalnej tablicy Taguchi L9

Process No.	Parameters and levels				Parameter value			
					Nitrogen pressure (Pa)	Arc current (A)	Bias voltage (V)	Substrate temperature (^{0}C)
1	A_1	B_1	C_1	D_1	0.8	50	-50	400
2	A_1	B_2	C_2	D_2	0.8	55	-75	430
3	A_1	B_3	C_3	D_3	0.8	65	-150	465
4	A_2	B_1	C_2	D_3	1.7	50	-75	465
5	A_2	B_2	C_3	D_1	1.7	55	-150	400
6	A_2	B_3	C_1	D_2	1.7	65	-50	430
7	A_3	B_1	C_3	D_2	2.6	50	-150	430
8	A_3	B_2	C_1	D_3	2.6	55	-50	465
9	A_3	B_3	C_2	D_1	2.6	65	-75	400

of the Berkovitch diamond indenter at a controlled maximum depth mode. The maximum indentation depths during all measurements were less than one-tenth of the coating thickness (300 nm). The adhesion strength of the coatings to the steel substrate was evaluated by scratching with use of a Revetest tester from CSM Instruments. The measurements were made at standard parameters, i.e., at loading rate dL/dt = 100 N/min and scratching speed v = 10 mm/min. For the quantitative adhesion evaluation the lowest value of the indenter load at which a specific damage occurred in a form of coatings spalling was assumed as the critical failure load L_{C2}. The high temperature tribological tests of the AlCrN coatings were carried out in ambient air at a temperature of 500°C using a ball-on-disk high temperature tribometer THT from CSM Instruments in an unlubricated mode at a friction a distance 100 m, along a circular track of 6 mm diameter, against ceramic balls from Al_3O_3 of 6 mm in diameter, at a normal load of 10 N and at a sliding speed of 0.1 m/s. For each specimen three separate tests were made. In order to calculate the the mean value of the volume wear three profiles of the bottom of the wear track in the direction perpendicular to the sliding motion were recorded with use of a Taylor Hobson profilometer.

According to the Taguchi method, the effect of the deposition process parameters on the coatings quality was evaluated from the changes in the average values of the signal-to-noise ratio (SNR) for any of the three levels of the deposition parameters. By comparing the effects of the SNRs values for each parameter onto the wear volume, the optimum experimental conditions for manufacturing AlCrN coatings with the highest wear resistance were established. In order to obtain the greatest resistance of the coating to the volume wear the optimization criterion "the smaller-the-better" was used in the present study.

RESULTS AND DISCUSSION

A summary of the properties for investigated AlCrN coatings is given **Table 3**. The results of the tribological tests executed by means of the ball-on-disc method indicated that, for all the investigated AlCrN coatings, the values of the friction coefficient (COF) in the steady-state phase of the runs remained practically constant at a level of 0.70±0.05, whereas the volume wear was changing with changes in deposition parameters (**Fig. 1**, **Tab. 3**). The lowest value of the volume wear (0.033 mm^3) was observed for the coating produced in the process no.7. Based on the statistical analysis (**Fig. 2**) the greatest resistance to wear of the AlCrN coatingwas obtained for the following combination of the deposition parameters $A_3B_1C_3D_3$: the nitrogen pressure 2.6 Pa, arc current 50 A, negative bias voltage 150 V and substrate temperature 465°C. It was proved with use of the Analysis of Variance (ANOVA) that

Table 3. Properties of AlCrN coatings (the mean values) obtained for 9 different sets of deposition parameters in the Taguchi L9 method

Tabela 3. Właściwości powłok AlCrN (wartości średnie) osadzonych przy 9 różnych zestawach parametrów wybranych wg metody Taguchi L9

Coating No.	Roughness R_a (μm)	Hardness H (GPa)	Young modulus E (GPa)	Adhesion strength L_{C2} (N)	Volume wear W_V (mm^3)	
1	0.31±0.04	21.5±2.4	268±32	28±1	0.097	±0.008
2	0.36±0.04	24.6±3.0	292±46	32±5	0.123	±0.024
3	0.46±0.04	21.0±2.5	286±26	15±1	0.108	±0.007
4	0.30±0.08	18.2±1.3	211±9	25±3	0.061	±0.069
5	0.23±0.04	24.6±2.4	273±31	15±9	0.053	±0.020
6	0.23±0.02	18.4±1.7	241±12	29±2	0.135	±0.026
7	0.15±0.02	27.6±3.0	328±49	23±2	0.033	±0.005
8	0.24±0.05	20.0±3.0	234±29	25±2	0.059	±0.022
9	0.19±0.04	19.3±1.9	263±32	25±3	0.092	±0.032

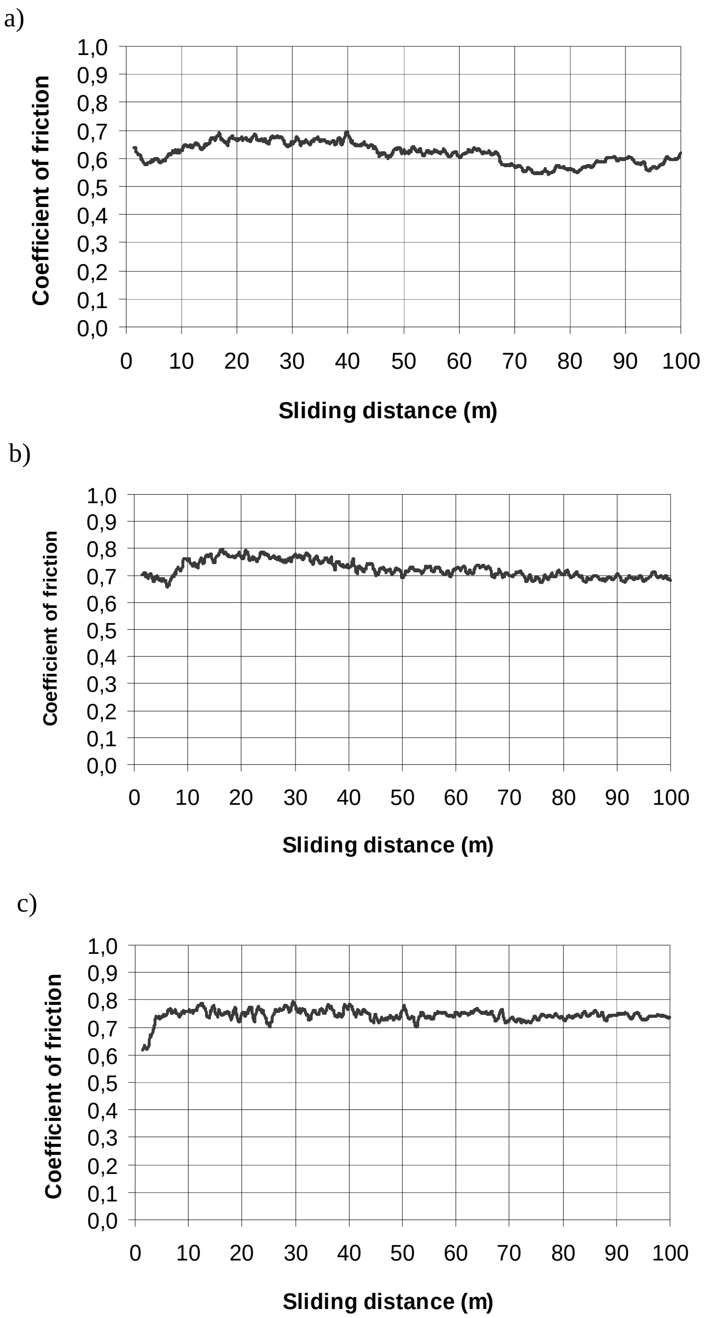


Fig. 1. Friction coefficients of the AlCrN coatings deposited in the processes: a) no. 3, b) no. 5 and c) no. 7

Rys. 1. Współczynniki tarcia zarejestrowane dla powłok AlCrN osadzonych w procesach: a) nr 3; b) nr 5 oraz c) nr 7

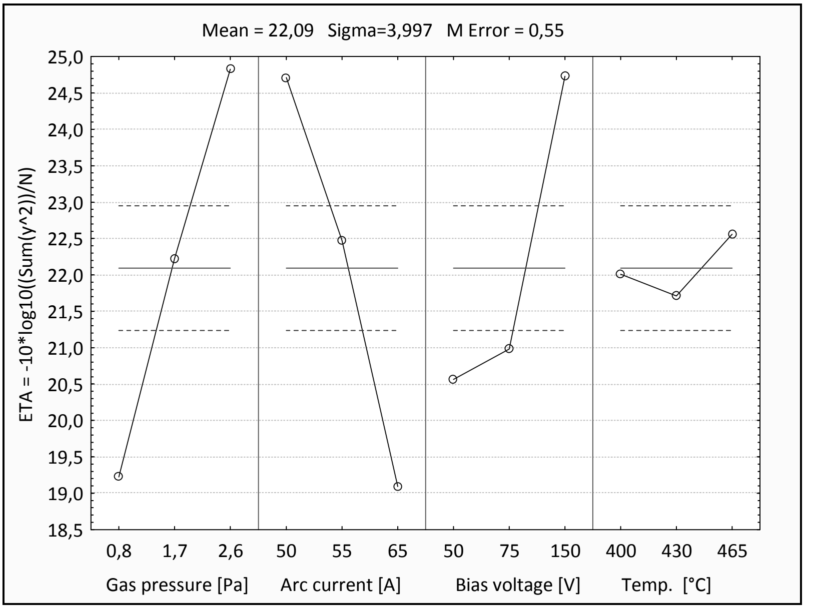


Fig. 2. The effects of each deposition parameter on the volume wear of the AlCrN coatings
Rys. 2. Wpływ poszczególnych parametrów osadzania na wartość objętościowego zużycia powłok AICrN

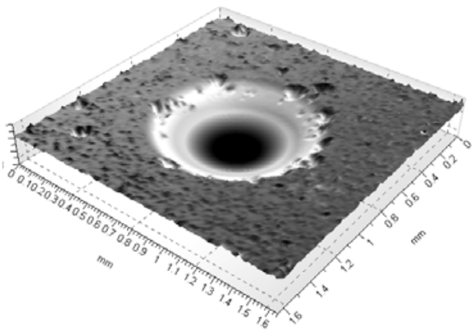
the relative contributions of the process parameters to minimise the volume wear of the investigated coatings were as follows: 43.21% for nitrogen pressure, 18.13% for arc current, 32.18% for bias voltage and 6.48% for temperature (**Tab. 4**). The results of the Taguchi analysis also indicate that the nitrogen pressure, the arc current and the bias voltage are the dominant parameters for obtaining the AlCrN coatings with a best of anti-wear resistance, whereas the substrate temperature did not have a significant effect on the wear volume of these coatings in the tested range 400°C–465°C.

Table 4. The results of ANOVA analysis of the effect of deposition parameters on the resistance to wear of the AlCrN coatings
Tabela 4. Wyniki analizy ANOVA wpływu parametrów osadzania na właściwości przeciwzużyciowe powłok AlCrN

Parameters		Sum of squares	Degree of freedom	Mean square	Probability	Contribution wear (%)
Nitrogen pressure (Pa)	A	47.14	2	23.57	0.023	43.21
Arc current (A)	B	47.98	2	23.99	0.022	18.13
Bias voltage (V)	C	31.59	2	15.80	0.034	32.18
Substrate temperature (°C)	D	1.10	2			6.48
Error		1.10	2	0.55		

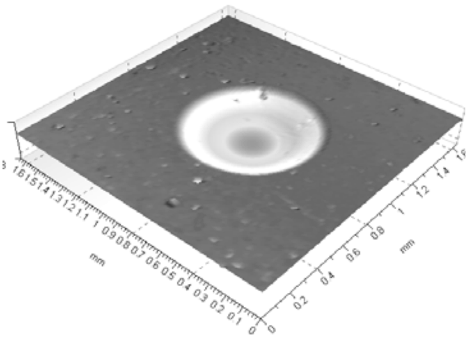
The investigations of the surface topography showed that the AlCrN coatings deposited by cathodic arc method have many defects such as: the microdroplets, the pinholes, the conical hillock anomalies and the shallow craters. The quantity and size of these defects depend on the deposition conditions and affect significantly not only the coatings morphology and surface roughness but also their mechanical and tribological properties (hardness, Young modulus, adhesion and wear resistance). It looks like that in comparison with other tested coatings the AlCrN one deposited in the process no.7 has the best properties, i.e., the greatest resistance to wear together with a low participation of the surface defects and a very dense structure (**Fig. 3**). The fact that the defects in the coating surface strongly influence tribological

a)



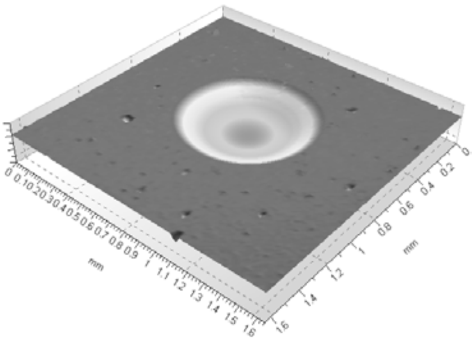
$R_a = 0.46\ \mu m$ $W_v = 0.106\ mm^3$

b)



$R_a = 0.23\ \mu m$ $W_v = 0.053\ mm^3$

c)



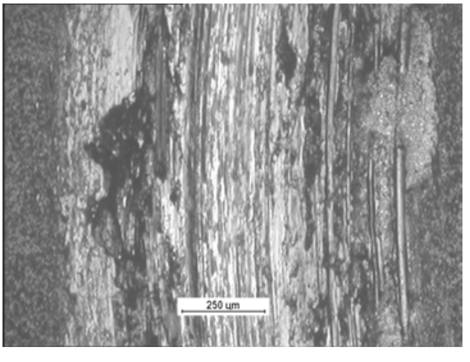
$R_a = 0.15\ \mu m$ $W_V = 0.032\ mm^3$

Fig. 3. Surface topographies and spherical cross sections of the AlCrTiN coatings made by ball-cratering method: (a) coating No. 3, (b) coating No. 5, (c) coating No. 7

Rys. 3. Topografia powierzchni i przekroje sferyczne powłok AICrN (wykonane metodą kulotestu): a) powłoki nr 3, b) powłoki nr 5, c) powłoki nr 7 performance of the tribosystem under dry sliding contact conditions is well known and presented in a number of scientific publications **[L. 16-17]**. The results of this study also confirm that in order to improve the tribological properties of PVD hard coatings it is important to minimize the concentration of the surface defects.

The microscopic examination of the inner area of the wear track of the AlCrN coatings revealed the occurrence of parallel grooves or scratches along the sliding direction, but their number and depth varied depending on deposition conditions of the coatings (**Fig. 4**). The study has shown as well that this kind of wear occurs as a result of the abrasive action of hard particles separated from the coating due to its delamination induced by high contact stress and promoted by defects present in the coating surface **[L. 18]**. The smooth surface (like a ground one) observed in the wear track of the coating no. 7 with the greatest resistance to the abrasive wear gives evidence for a good resistance to the spalling and the optimal mechanical properties of this coating (with a highest hardness 27.6±3.0 GPa as well as a good adhesion to the substrate 23±2 N).

a) b)



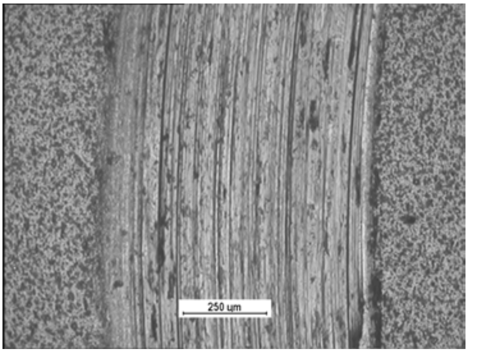


Fig. 4. Wear track morphology of the AlCrN coatings: a) coating no. 6; b) coating no. 7
Rys. 4. Morfologia śladu zużycia powłok AlCrN: a) powłoki nr 6; b) powłoki nr 7

Having established optimal process parameters for deposition of AlCrN coating with the greatest resistance to wear further analysis was conducted in order to check the accuracy of Taguchi analysis. To this end three substrates were coated with use of the set of the optimal process parameters. The results of the verification test are shown in **Tab. 5.** These results demonstrate good compliance between the predicted and measured resistance to wear of the AlCrN coating and distinctly indicate that the Taguchi method delivers reliable means for optimization of tribological properties of the hard PVD coatings.

Table 5. Results of the verification test
Tabela 5. Wyniki testu weryfikacyjnego

Deposition parameters	Hardness H (GPa)	Young modulus E (GPa)	Adhesion strength L_{C2} (N)	Volume wear W_V (mm^3)	
Initial experiment parameters A3B1D3D2	27.6±3.0	328±49	23±2	0.033	±0.005
Parameters of verification experiment A3B1C3D3	25.3±3.0	278±21	26±1	0.032	±0.004

CONCLUSIONS

In this study the Taguchi method was adopted to establish process parameters for cathodic arc deposition of AlCrN coating with the highest resistance to wear. Based on the statistical analysis the dominant deposition parameters affecting the coating resistance to wear were determined. The optimum process conditions necessary to achieve the highest wear resistance for AlCrN coating in combination with good mechanical properties were established as well.

The following conclusions can be drawn from the investigations conducted in the work:

- Defects in the coating surface strongly influence tribological behaviour of the tribosystem under sliding contact conditions and hinders in achievement of high resistance to wear, therefore the AlCrN coating must have suitable hardness and adhesion and a smooth surface with very limited number of defects,.
- The most important process parameters for improvement of mechanical and tribological properties of AlCrN coatings are the nitrogen pressure, arc current and substrate bias voltage.
- The optimum deposition conditions to achieve the greatest resistance to wear of the AlCrN coatings are as follows: the nitrogen pressure 2.6 Pa, arc current 50 A, negative bias voltage 150 V and temperature 465°C.
- The Taguchi method may be successfully used for establishing optimum parameters of the cathodic arc process allowing deposition of the AlCrN coatings with improved tribological behaviour.

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Streszczenie

W artykule zaprezentowano wykorzystanie metody Taguchi do optymalizacji odporności przeciwzużyciowej twardych powłok AlCrN wytwarzanych metodą katodowego odparowywania łukowego. Do wyznaczenia wpływu warunków osadzania na zdefektowanie powłok, ich właściwości mechaniczne oraz tribologiczne wytypowano cztery podstawowe parametry procesu osadzania, tj.: ciśnienie azotu, prąd łuku, napięcie polaryzacji i temperaturę podłoża z trzema poziomami wartości. Na podstawie wykonanych analiz statystycznych określono optymalne parametry osadzania umożliwiające wytwarzanie powłok AlCrN o największej odporności na zużycie. Uzyskane wyniki badań wskazują, że spośród analizowanych parametrów procesu osadzania dominujący wpływ na zminimalizowanie zużycia objętościowego wywierają: ciśnienie azotu, prąd łuku, napięcie polaryzacji. Badania topografii powierzchni i właściwości mechanicznych wytworzonych powłok CrAlN wykazały, że dla zapewnienia najwyższej odporności na ścieranie powłoka powinna charakteryzować się wysoką gładkością powierzchni (z niskim udziałem wad powierzchniowych), dostatecznie dużą twardością oraz dobrą przyczepnością do podłoża. Dla osiągnięcia podwyższonej odporności na zużycie dla powłoki AlCrN wyznaczono optymalne warunki osadzania, które powinny być następujące: ciśnienie azotu 2,6 Pa, prąd łuku 50 A, ujemne napięcia polaryzacji 150 V i temperatura podłoża 465°C.