Mechanical and anti-wear properties of multi-module Cr/CrN coatings

Łukasz Szparaga*, Katarzyna Mydłowska, Adam Gilewicz and Jerzy Ratajski

Department of Technology and Education,
Koszalin University of Technology,
ul. Śniadeckich 2, 75-453 Koszalin, Poland
Email: lukasz.szparaga@tu.koszalin.pl
Email: katarzyna.mydlowska@tu.koszalin.pl
Email: adam.gilewicz@tu.koszalin.pl
Email: jerzy.ratajski@tu.koszalin.pl
*Corresponding author

Abstract: The objects of investigations were Cr/CrN multi-module coatings deposited using a cathodic arc evaporation method (CAPVD) on HS6-5-2C steel used as a substrates. Analysed coatings possesses seven Cr/CrN modules of fixed thickness each, with various thicknesses of Cr and CrN layers. Aiming for the evaluation of mechanical properties of tested multi-module Cr/CrN coatings, its hardness and Young's modulus were measured, on the basis of which were determined values of H/E and H^3/E^2 ratios. Coatings wear and friction coefficients were measured in so called ball-on-disc test. The adhesion of the coatings was evaluated using scratch tester and was shown that main mechanism of adhesive damage of all tested coatings at higher loads are buckle spallations. All tested coatings are also characterised by good adhesion to the substrate, which is evidenced by the fact that cracked coating remains inside the scratch track. Basing on the analysis of obtained experimental results it was confirmed and explicitly shown that the thickness of the individual layers of Cr and CrN in the multi-module coating significantly affects its critical loads (in scratch test), fracture toughness and wear rate.

Keywords: thin hard coatings; physical vapour deposition; PVD; anti-wear coatings; multi-layer coatings; nanoindentation; adhesion; scratch test; fracture toughness; ball-on-disc test; hard chromium replacement.

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Biographical notes: Łukasz Szparaga is a Lecturer at the Faculty of Technology and Education of Koszalin University of Technology. His scientific work deals with issues related to surface engineering and PVD coatings, mainly in the area of FEM numerical simulations.

Katarzyna Mydłowska is a PhD student and an Assistant at the Faculty of Technology and Education of Koszalin University of Technology. Her scientific work deals with issues related to surface engineering, PVD coatings, duplex technologies and thermal analysis.

Adam Gilewicz is a Technical and Scientific Specialist at the Faculty of Technology and Education of Koszalin University of Technology. He received his PhD in the field of surface engineering regarding anti-wear PVD coatings. He is an expert in the field of PVD coating deposition technologies.

Jerzy Ratajski is a Professor at the Faculty of Technology and Education of Koszalin University of Technology. He is the Head of the Department of Biomedical Engineering. He is an expert in the field of thermo-chemical surface treatment, mainly nitriding. His current scientific work focuses on anti-wear PVD coatings and duplex technology for surface modification of materials.

1 Introduction

The electroplated chromium is used for decades for producing a relatively thick, and hard, anti-wear coatings in a variety of industries – including automotive, aircraft and aerospace industries. However, the chemicals used for the deposition of these coatings contain hexavalent chromium (Cr6+ ions) and chromium in this state is extremely toxic. Therefore, due to the European directive concerning chemicals (REACH), hard chrome coatings, produced in toxic and carcinogenic baths, must be replaced by 'green' solutions. In these industries, in some applications, for example for the parts of an aircraft engine, coatings produced by physical vapour deposition (PVD) methods may be an alternative to hard chrome coatings (Bielawski, 1998; Bielawski et al., 2002; Hetmańczyk et al., 2007).

Particular interest in this area is directed towards the coating on the base of chromium nitride. These coatings are, among other, characterised by relatively low value of residual stress and good resistance to oxidation at high temperatures (Gauzzi et al., 2008; Lee et al., 2001; Mydłowska et al., 2016). For monolayer coatings deposited by PVD techniques, it is often observed that increasing the hardness of the coatings is accompanied by a simultaneous increase in the Young's modulus and decrease of their ductility (impact resistance), which leads to a reduction of coating's fracture toughness due to the reduction of their ability to dissipate mechanical energy (through plastic deformation). In order to limit these effects, for example, a more complex structure of the coating is used such as multilayer coatings and multi-module Cr/CrN (Marulanda et al., 2012; Polcar et al., 2009; Ratajski et al., 2015), CrN/CrCN (Gilewicz et al., 2015; Szparaga et al., 2015a, 2015b) or gradient CrN/Cr(C)N coatings (Fuentesa et al., 2008; Ratajski and Szparaga, 2012; Szparaga et al., 2015c; Zhang et al., 2009). The such structure of coating increases the resistance to cracking and enhances the adhesion state, e.g., by reducing residual stresses in the coating.

The type of coating on Cr/CrN base is also known as a intelligent coating because their structure, among other Cr to CrN thickness ratio in multi-module Cr/CrN coating, determines their mechanical properties and the intensity of wear depending on the operating conditions (Martinez et al., 2003; Mazurkiewicz et al., 2015; Wieciński et al., 2011, 2014, 2015). In such combination the ductile phase (Cr) is capable of absorbing excessive plastic deformation, while the hard phase (CrN) provides the resistance to wear.

For nearly three decades in designing of structure of multi-layer, monolayer and multi-module coatings a great role play finite element method (FEM) simulations (Dobrzański et al., 2007; Pakuła et al., 2016; Ratajski and Szparaga, 2012; Szparaga et al., 2015a, 2015c; Śliwa et al., 2016; Śliwa, 2017; Zukowska et al., 2016). Among other FEM model was used in optimisation procedure for eight-layer Cr/CrN coating on A2 steel substrate in terms of mechanical properties (Lakkaraju et al., 2006). In this model, the decisional variables were individual thicknesses of the layers. The decisional criteria were the values of strain discontinuities at the coating/substrate interface and stresses in the outer CrN layer generated by combined normal and tangential mechanical loads. The result of the simulation was optimal architecture consisting of Cr/CrN modules of different thicknesses, i.e., the thinnest possible metallic Cr layer at substrate/coating interface and thickest CrN outer layer. In our research (Szparaga et al., 2015a), we have developed the optimisation procedure supporting the prototyping of the geometry of multi-module CrN/CrCN coatings, deposited on 42CrMo4 steel substrates, in terms of mechanical properties. Decisional criteria were the functions of the state of residual stress and strain in the coating/substrate system, generated by external mechanical loads. The main result was a classification of obtained Pareto-optimal solutions (optimal coating structure – number of modules, thicknesses of Cr, CrN and CrCN layers) which allow grouping them into classes including potential instability of the technological parameters of coating deposition processes and similar mechanical properties.

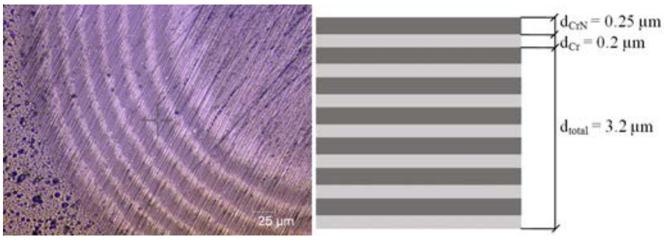
In summary, the successful design of wear-resistant coatings requires consideration of many factors, including mechanical and residual stresses in the system, coating-substrate adhesion as well as hardness and fracture toughness of each layer. In the case of multi-module Cr/CrN coatings, as already mentioned, it is particularly important to choose a ratio between the thickness of the Cr layers and the thickness of the CrN layers. When it is properly selected, the interfacial stress is reduced as well as the propagation of cracks across the coating is limited. However, when the proportion of metallic layers in the coating is too high, the resistance to wear is rapidly degraded.

In this paper, following by the guidelines regarding the architecture of coatings resulting from the design of multilayer coatings using FEM, tests of fracture toughness, wear resistance and adhesion of multi-module Cr/CrN coatings with diametrically different architecture deposited on HS6-5-2C steel substrates were carried out. Selection of coating architecture was dictated by the assessment of the possibilities of work of such steel substrate/CrN/Cr coating systems in very diverse operating conditions, mainly at varied external load.

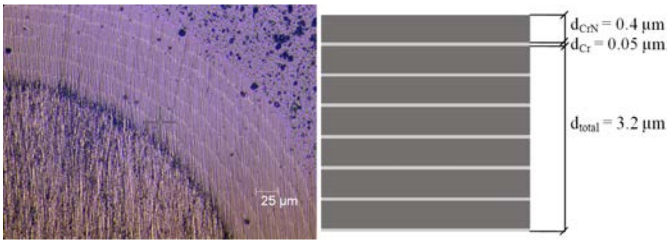
2 Object and research methodology

The objects of investigations were Cr/CrN multi-module coatings with different structure of modules – marked as A–D types (Figure 1). Each type of analysed coatings possesses seven modules with fixed thickness of 0.45 μm but with various thicknesses of Cr and CrN layers. Coatings of types A and B are composed of identical modules with Cr to CrN thicknesses ratio respectively equal to 1:1 and 1:8. The structure of type C coating is almost the same as type B but in the third module (counting from the surface) the thickness ratio of Cr to CrN is equal to 1:1. The aim of this modification was to enhance the ability of stress reduction inside coating below contact surface. Coatings of type D are composed of two modules with Cr to CrN thicknesses ratio equal to 1:8 and five modules with Cr to CrN thicknesses ratio 8:1. Two hard upper modules will provide good wear resistance and lower softer modules will dissipate energy of residual stresses. It is also expected that type D coatings would posses good fracture toughness due to a smaller difference in the hardness between the coating and the substrate.

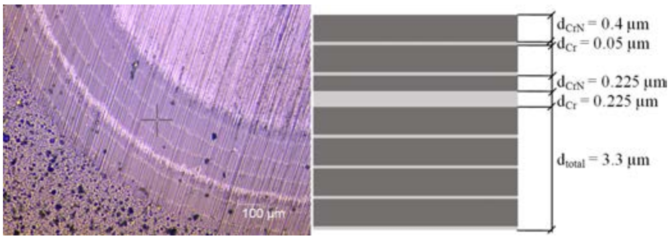
Figure 1 The structure of the tested multi-module Cr/CrN – coatings scheme and microscopic photograph after Calotest, (a) type A (b) type B (c) type C (d) type D (see online version for colours)



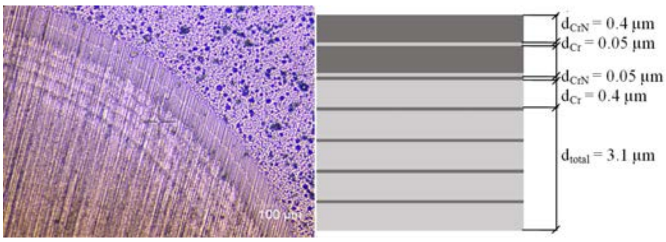
(a)



(b)



(c)



(d)

The multi-module Cr/CrN coatings were deposited using a cathodic arc evaporation method (CAPVD) on HS6-5-2C steel used as a substrates. For deposition was used device which allows for work with six sources and with two reactive gases simultaneously. During multi-module Cr/CrN coatings deposition two targets made of pure chromium were used. Prior to deposition process substrates were grinded, polished and ultrasonically cleaned. In a working chamber, they were also heated for 1.5 h to about 350°C and subjected to ion surface cleaning for 10 min at working gas (Ar) pressure of 0.5 Pa and substrate bias voltage –600 V. Then the arc discharge was initiated and substrate bias voltage was gradually reduced to –70 V, what resulted producing on the substrate surface Cr layer with a thickness of approx. 0.1 μm in order to enhance the adhesion of the coating. Bias voltage of the substrate during deposition of multi-module coatings was –70 V, and arc current was 80 A. The chromium layer was obtained at an argon pressure in the working chamber equal to 0.5 Pa. CrN layer was obtained by introducing the N_2 to the atmosphere in the working chamber. The gas pressure was 1.8 Pa. The varied thickness of the layers in the individual modules according to the designed A-D architectures were obtained by changing the deposition time.

Nanoindentaion measurements were performed using FISCHERSCOPE HM2000 XYp. System was equipped with diamond Berkovich indenter. Nanoindentation curves were determined for fixed penetration depth of the indenter equal to 0.3 μm. For each of the samples were recorded 15 curves and one process of indentation lasted about 90 s. In order to assess the mechanical properties of the coatings, curves, for which determined hardness was closest to the average hardness values of all the recorded curves, were selected.

Coatings wear and friction were measured using T-10 ball-on-disc tribometer (produced by the Institute for Sustainable Technologies – National Research Institute, Radom, Poland). As a counterspecimen was used alumina ball with a diameter 10 mm and applied load was 20 N. Wear was conducted at room temperature and constant rotational speed of 159 rpm. A sliding distance was 2,000 m for coatings B and D, 1,500 m for type C and 200 m for type A of coating. The volumetric wear rate of the coating was calculated on the basis of the cross profile of wear track designated by profilograph HOMMEL TESTER T8000.

The adhesion of the coatings was evaluated using scratch tester (REVETEST®) with a diamond stylus with radius of 0.2 mm. The maximum load was 150 N and scratch distance was equal to 10 mm. During the test were registered values of normal force, friction force and acoustic emission as a function of scratch distance. Based on these curves and microscopic images of scratch, values of two critical loads were determined. First critical load (Lc_1) is a load in which first cracks in a coating are initiated, whilst second (Lc_2) is a load in which coatings total delamination occurs.

3 Experimental results

Figure 2 shows values of hardness of the tested coatings, determined based on the results of nanoindentation tests (Figure 3), at a specified displacement of indenter equal to 0.3 μm.

Figure 2 The Vickers hardness of the tested multi-module Cr/CrN coatings determined from the nanoindentation curves (see online version for colours)

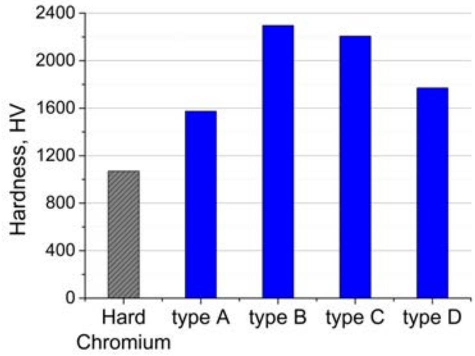


Figure 3 Nanoindentation load-displacement curves for tested coatings of types A–D (see online version for colours)

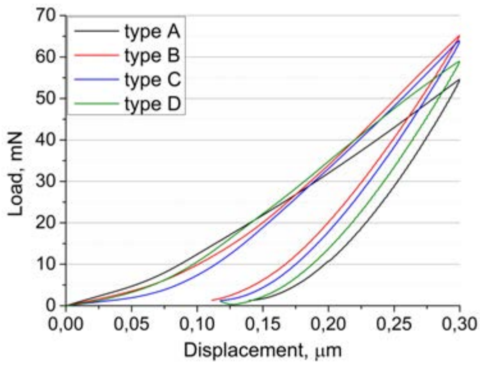


Table 1 The mechanical properties of the coatings of types A–D determined from the indentation curves

	H [GPa]	*E [GPa]*	W_e *[%]*	H^3/E^2	*H/E*
Type A	15.05	272.1	45.7	0.046	0.0553
Type B	22.1	265.5	65.2	0.153	0.0832
Type C	21.5	274.2	64.0	0.132	0.0784
Type D pure Cr	17.51	276	51.6	0.07	0.0634
Hard chromium	10.0	235		0.018	0.0426

The objects of investigations, presented in the article, were Cr/CrN multi-module coatings with different structure of modules. Thus, in the measured hardness values the top layers of the coatings are of major importance. However, for each of the tested multi-module coatings, hardness (Figure 2) significantly exceed the values that characterise commercially used anti-wear electrodeposited coatings of hard chromium which hardness is up to 1,200 HV (Sohi et al., 2003). As expected, the highest hardness characterises coatings of B and C types due to the large proportion of hard CrN phase in the coating volume. Also, the top two modules in the coating of D type, with a thickness of approx. 0.9 μm, is a zone of increased hardness due to the relatively high CrN/Cr thickness ratio. Therefore, the coating D is characterised by higher hardness than A type of coating, despite larger content of the soft Cr phase in the whole coating volume.

On the basis of the analysis of the conducted tests, it follows that the Young's modulus (E) of all A–D coatings are within a relatively narrow range (Table 1). However, the coating B, in which the largest share is hard layers (CrN), is characterised by the smallest value of this parameter, coating of B type is also characterised by the largest H/E, H^3/E^2 ratios and value of W_e (elastic recovery) indicator. The higher value of these indicators means higher resistance to a fracture toughness and higher resistance to plastic deformation of the coating (Leyland and Matthews, 2000; Musil et al., 2012a, 2012b; Wang et al., 2015, 2016, 2017a, 2017b, 2018), what may indicate a greater ability of the coating to carry higher mechanical loads. In turn, coating A is characterised by the lowest values of these indicators, i.e., according to the above interpretation, this coating has the highest susceptibility to plastic deformation.

The difference in the ratio of layers thicknesses in each module also has a significant influence on the adhesion of the coating as demonstrated by values of critical loads defined by scratch test, shown in a Figures 4 and 5.

Figure 4 The values of the critical loads Lc_1 and Lc_2 determined on the basis of scratch test for coatings of A–D types (see online version for colours)

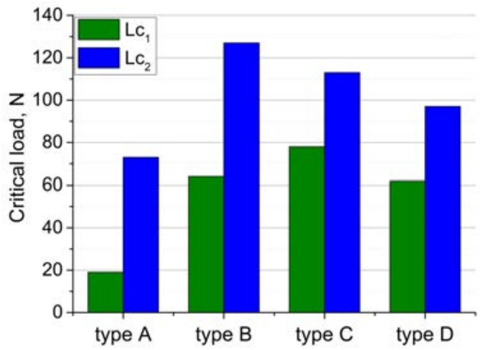
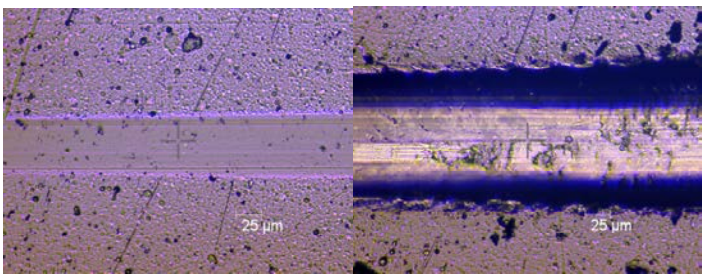


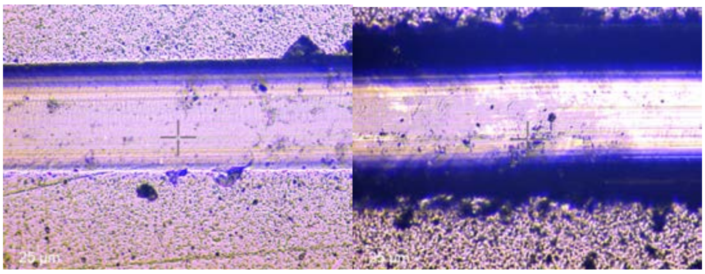
Figure 5 Photographs of the scratch track in points corresponding to Lc_1 and Lc_2 for A type of coating (see online version for colours)



(a) (b)

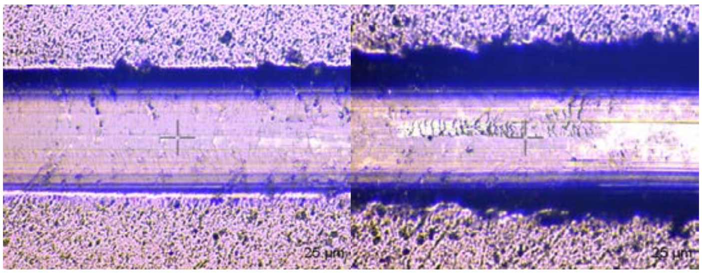
All tested samples are characterised by high resistance to total delamination of the coating, which determines value of second critical load Lc_2, however, this load for the best coating in this set (type B) is more than 1.5 times higher than for the worst coating (type A). From the viewpoint of operating conditions of machine parts covered by anti-wear coatings, more important parameter is first critical load Lc_1, wherein the first cracks and spallation of the coating occur. The highest value of this load characterises a coating of type C (78 N), whereas, the lowest one, as is the case of Lc_2, type A of coating (19 N). Similar values of Lc_1 for B and D coatings (64 and 62 N respectively) indicate their high resistance to crack initiation (Figure 5). Tracks after scratch test for studied systems A–D reveal different mechanisms of coatings degradation, types of cracks and its locations.

Figure 6 Photographs of the scratch track in points corresponding to Lc_1 and Lc_2 for B type of coating (see online version for colours)



(a) (b)

Figure 7 Photographs of the scratch track in points corresponding to Lc_1 and Lc_2 for C type of coating (see online version for colours)



(a) (b)

The main mechanism of damage of the coatings of types B and C are at low loads tensile cracks, which is characteristic for brittle coatings having higher hardness than the substrate. The same type of damage is also presented in the case of A type of coating, however, in contrast to the coatings B and C, is not accompanied by the so-called shell cracks appearing on the edges of track resulting from the accumulation of stresses on the scratch edges. In the case of D type of coatings, at a lower loads are observed conformal cracks, which demonstrate a greater ductility of the coating. The main mechanisms of adhesive damage of all tested coatings at higher loads are buckle spallations. Good adhesion of all tested coatings is evidenced by the fact that cracked coating remains inside the scratch track.

Figure 8 Photographs of the scratch track in points corresponding to Lc_1 and Lc_2 for D type of coating (see online version for colours)

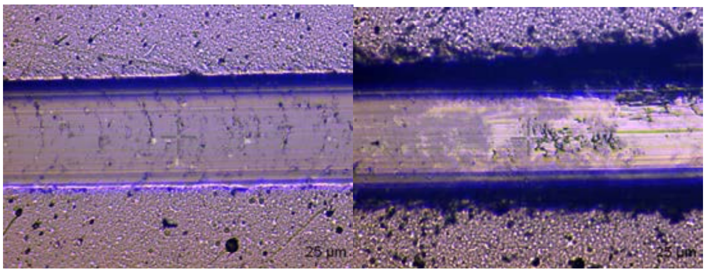


Figure 9 Dependence of friction coefficient as a function of sliding distance during ball-on-disc tribotest for coatings of types A–D (see online version for colours)

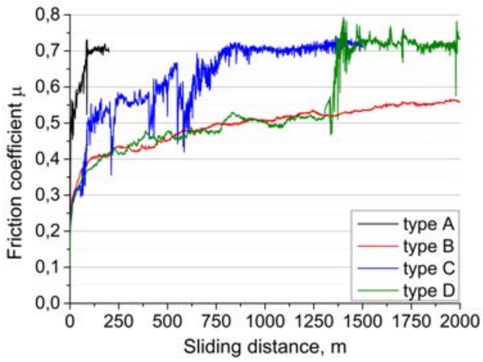


Figure 10 Volumetric wear rate of coatings of A–D types determined after ball-on-disc tribotest (see online version for colours)

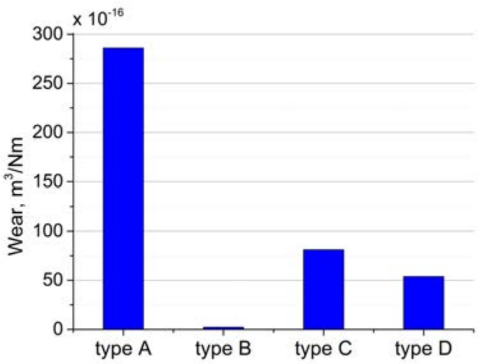


Figure 9 shows dependence between friction coefficient and sliding distance during ball-on-disc tribotest. The wear rate, which is the ratio of the volume of worn material of coating to sliding distance, calculated using cross profile of wear track after the test. Values of volumetric wear ratio are shown in Figure 10. It can be clearly seen that the best anti-wear properties under adopted test conditions characterise a coating with Cr to CrN thickness ratio equal to 1:8 (coating B).

For coatings of B type (Figure 9) is observed the monotonous increase in the friction coefficient during the test, which provides uniform coatings degradation over time. For the coating of D type is also observed uniform wear over a distance of approximately 1,300 m followed by a rapid total removal/severe damage of coating revealed by sudden nonlinear and non-monotonic change in the friction coefficient. Wear of type A coating is progressing also in the linear trend, but the total wear of coating occurred at a distance of less than 200 m. Whereas, the coating of type C shows the completely different nature of wear in comparison with other types of tested multi-module coatings. Namely, the increase in the friction coefficient during the test is highly nonlinear, which may indicate a sudden damage of coating in the form of cracks and chipping of material. Complete removal of the coating of type C occurs after the distance about 800 m.

The largest volume loss of material in the wear track is observed for type A of coating (having a thickness of Cr and CrN layers in each module 0.2 μm and 0.25 μm respectively). Wear of the coating in this case is higher by more than two orders of magnitude than wear of a coating of type B. Wears of C and D coatings is apparently higher than the type B of coating ($8.1 \cdot 10^{-15}$ and $5.4 \cdot 10^{-15}$ [m^3/N·m] respectively), however, its values can still be described as very low. As an example, in Voevodin et al. (1999) wear rate of nanocomposite anti-wear coatings for aircraft and space applications, defined by pin-on-disc test with a similar contact pressure (approx. 1.6 GPa) was 1 to $9 \cdot 10^{-15}$ [m^3/N·m] which, according to authors, is highly satisfactory result.

Relatively high resistance to the crack initiation of type C of coating (high critical load Lc_1) as well as resistance to initiation and growth of cracks demonstrated by coating D (lower hardness and relatively high value of Lc_1), in combination with a low wear rate for both coatings, makes them potentially good candidates for anti-wear coatings deposited on the elements used in the aerospace industry, which operate at lower contact pressures than used in presented studies.

4 Conclusions

Basing on the analysis of obtained experimental results it was confirmed that the thickness of the individual layers of Cr and CrN in the multi-module coating significantly affects its critical loads (in scratch test), fracture toughness and wear rate.

In particular:

- All tested coatings are characterised by high resistance to total delamination, which determines value of second critical load Lc_2.
- The type B (CrN to Cr thickness ratio in each module equal to 8:1) coating, which is characterised by the highest hardness, the highest H/E and H^3/E^2 ratios and the highest Lc_2 load value, is characterised by the lowest wear of all tested coatings.
- The highest value of the first critical load Lc_1, wherein, the first cracks and spallation of the coating occur, is characterised by coating type C (78 N). Slightly lower values of Lc_1 for B and D coatings (64 and 62 N, respectively) also indicate their high resistance to crack initiation, whereas, the lowest one, by type A of coating (19 N).

- In the coating of type C, through the change of the thickness of the layers in one of the modules compared with B coating, was obtained a slight reduction in coating hardness and resistance to total delamination, while simultaneous increment of Lc_1 value. This coating also showed relatively low wear rate at high contact pressure but the increase in the friction coefficient during the test is highly nonlinear, which may indicate a sudden damage of coating in the form of cracks and chipping of material.
- At low contact pressures, D type of coating with high content of pure Cr can be also characterised by very good anti-wear properties. Hardness lower of about 400 HV compared with type C of coating, may result during exploitation in smaller susceptibility to the growth of existing cracks in the coating. D type of coating is also characterised by uniform wear and high sliding distance before total removal in ball-on-disk test. It is therefore characterised by best mechanical integrity among the tested multi-module coatings, which makes it a potential candidate for anti-wear applications with lower contact pressures.

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