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Structure and properties of multicomponent coatings deposited onto sialon tool ceramics

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ABSTRACT

Purpose: The aim of this paper is to investigate structure, mechanical and functional properties of sialon tool ceramics with wear resistant multicomponent coatings deposited with PVD method.
Design/methodology/approach: The structural investigation includes the metallographic analysis on the scanning electron microscope. Examinations of the chemical compositions of the deposited coatings were carried out using the X-ray energy dispersive spectrograph EDS, and glow-discharge optical emission spectroscope GDOS. The investigation includes also analysis of the mechanical and functional properties of the material: microhardness tests of the deposited coatings, surface roughness tests, evaluation of the adhesion of the deposited coatings and tribological test made with the „pin-on-disk".

Findings: Deposition of the multicomponent coatings with the PVD method, on tools made from sialon's ceramics, results in the increase of mechanical properties in comparison with uncoated tool materials, deciding thus the improvement of their working properties.

Practical implications: The multicomponent coating carried out on multi point inserts (made on sintered sialon's ceramics) can be used in the pro-ecological dry cutting processes without using cutting fluids. However, application of this coating to cover sialon ceramics demands still both elaborating and improvement adhesion to substrates in order to introduce these to industrial applications.

Originality/value: The paper presents some researches of multicomponent coatings deposited by PVD method on sialon tool ceramics.

Keywords: Thin and hard coatings, Tool materials; PVD; Multicomponent coatings

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

A permanent aim of tool material designers for at least several decades was the invention an ideal tool material which would show a high ductility and a maximum possible resistance to wear in working conditions. Normally such a combination is impossible to obtain. Thus there have been various attempts made to solve, at least partly, the problem by creating layer structures using, among others, methods of thermo chemical treatment, by making composite materials and coating using CVD and PVD methods. On the one hand, among many grades of tool materials an sintered carbides has still been a part of material group about the biggest meaning in machining technologies. Beneficial utilitarian properties and an higher harden ability in comparison with a high speed steel and their low price follows from low manufacturing costs decide about their general application. Moreover, modern sintering methods let manufacture edges from sintered carbides about fine grains and better properties in comparison with carbides manufactured by standard methods. On the other hand a tool ceramic, and as well worked out before at the end of XX century a ß-sialon ceramic, gain bigger and bigger meaning in machining processes. The mechanical properties of this alloy ceramic are inherited from isomorphous ß-Si_3N_4, whereas chemical properties respond to aluminum oxide Al_2O_3. The application of physical vapour deposition PVD for the acquisition of multicomponent coatings of high wear resistance, also in high temperatures, enables to improve the properties of these materials in machining conditions, among others through the reduction of friction factor, rise of microhardness, improvement of tribological contact conditions in the contact area tool-machined item, and also to protect these materials against adhesive or diffusive wear and against oxidation [1-33].

The goal of this work is investigation of structure and properties of the PVD coatings deposited onto the sialon tool ceramics substrate.

2. Materials

The researches were done on multicomponent (Ti,Al)N and (Al,Cr)N coatings and multilayer and multicomponent (Al,Cr)N+(Ti,Al)N, (Ti,Al)N+(Al,Cr)N coatings on substrates from sintered tool materials with a special taking into account sialon ceramic. Substrate was prepared before a deposition process. The substrates were ultrasonic cleaned in a pure acetone and exsiccated in the dry heat stream. Coating were deposited by cathode arc evaporation method in a the PVD process.

3. Research methodology

Observation of both coatings structure and surface topography was carried out by using the ZEISS SUPRA 35 scanning electron microscopes. To obtain the topography and fracture images the Secondary Electrons (SE) technique were used with the accelerating voltage of 20 kV. The fractures were deposited a thin gold layer in purpose to carry away an electric charge from non-conductor substrate of sialon tool ceramics with investigated coatings. To chemical analysis was used the Energy Dispersive Spectrometry (EDS) method utilized detector OXFORD firm.

Changes of the chemical concentrations of the coating constituents in the direction perpendicular to its surface and of concentrations in the interlayer between the coating and the substrate material were evaluated basing on examinations made in the Leco Instruments glow-discharge optical emission spectroscope GDOS-850A. The following spectrometer Grimm lamp working conditions were determined during the examinations:

- lamp internal diameter – 4 mm;
- lamp power supply voltage – 700 V;
- lamp current – 20 mA;
- working pressure – 100 Pa.

The hardness of investigated coatings was measured into a dynamic ultra-microhardness testtester DUH 202 produced by Shimadzu company. During tests was used an Vickers indenter. Method allows onto researches of hardness a very thin coatings and foils without simultaneous their puncture. There was applied 50 mN loading, what completed that depth performed impression did not exceed 0,1 thickness of coatings, eliminated in this way the influence substrate onto a result of mensuration.

Adhesions of deposited coatings to the substrate was examined by Scratch Test method, using CSEM Revetest device. Diamond indenter was shifted on surface coating with load increasing from 0 to 200 N on the distance 10 mm. The load was increasing with a rate (dL/dt) 100 N/min. The critical load, which is a measure of adhesion, was determined on the basis of acoustic emission level (AE) and a observation of failure at the scratch on scanning electron microscopy DSM 940 from Opton corporation.

The investigations of surface roughness samples without coatings and deposited by studied coatings were measured on the Surtronic+3 profilometr produced by Taylor Hobos company. Investigation were made on gauge length of a test piece Lc = 0.8 mm with an accuracy of ±0.02 µm. The R_a parameter was accepted as quantity describe a roughness.

Tribological tests were carried out on the CSEM „pin-on-disk” tester in the following conditions: counter-specimen – ball made from the WC titanium carbide with the 6 mm diameter, counter-specimen load – 5 N, friction radius – 5 mm, linear velocity – 0.1 m/sec, ambient temperature – 20°C. The character of the developed failure was evaluated basing on observations on the light microscope and on the scanning electron microscope.

4. Discussion of investigation results

As the result of fracture coating observation onto scanning electron microscope it was found that all investigated coatings are characterized by compact fibrous structure without pores and well adhere to a substrate, furthermore particular layers coating of (Al,Cr)N+(Ti,Al)N adhere tight to each other. It was found that investigated coatings obtain by PVD technique is characterized a fine granular structure, which is corresponded to a IV (T) zone according to the Thornton model (Fig. 1). Observations of surface topography in a scanning electron microscope were found that a surface coatings morphology produced using the PVD technique onto sialon tool ceramic characterizes a considerable inhomogeneity connected with occurring numerous macromolecules in a drop shape and agglomerate formed in a consequence of joined few macromolecules (Fig. 2). Occurrence of these morphologic defects is connected with a cathode arc evaporation process. All these inhomogeneity observed in SEM cause an increase of roughness surface in comparison with uncoated inserts (Table 1).

Table 1.
Characteristics of the PVD coatings deposited on the sialon tool ceramics

Coatings	Thickness of coating, µm	Hardness HV0.5	Roughness factor R_a, µm	Critical loading L_c, N (max load, N)
-	-	1838	0.06	-
(Al,Cr)N	4.7	2235	0.33	53 (100)
(Ti,Al)N	5.2	2978	0.29	21 (100)
(Al,Cr)N+(Ti,Al)N	3.9	2558	0.44	69 (100)
(Ti,Al)N+(Al,Cr)N	4,0	3120	0,45	122 (200)

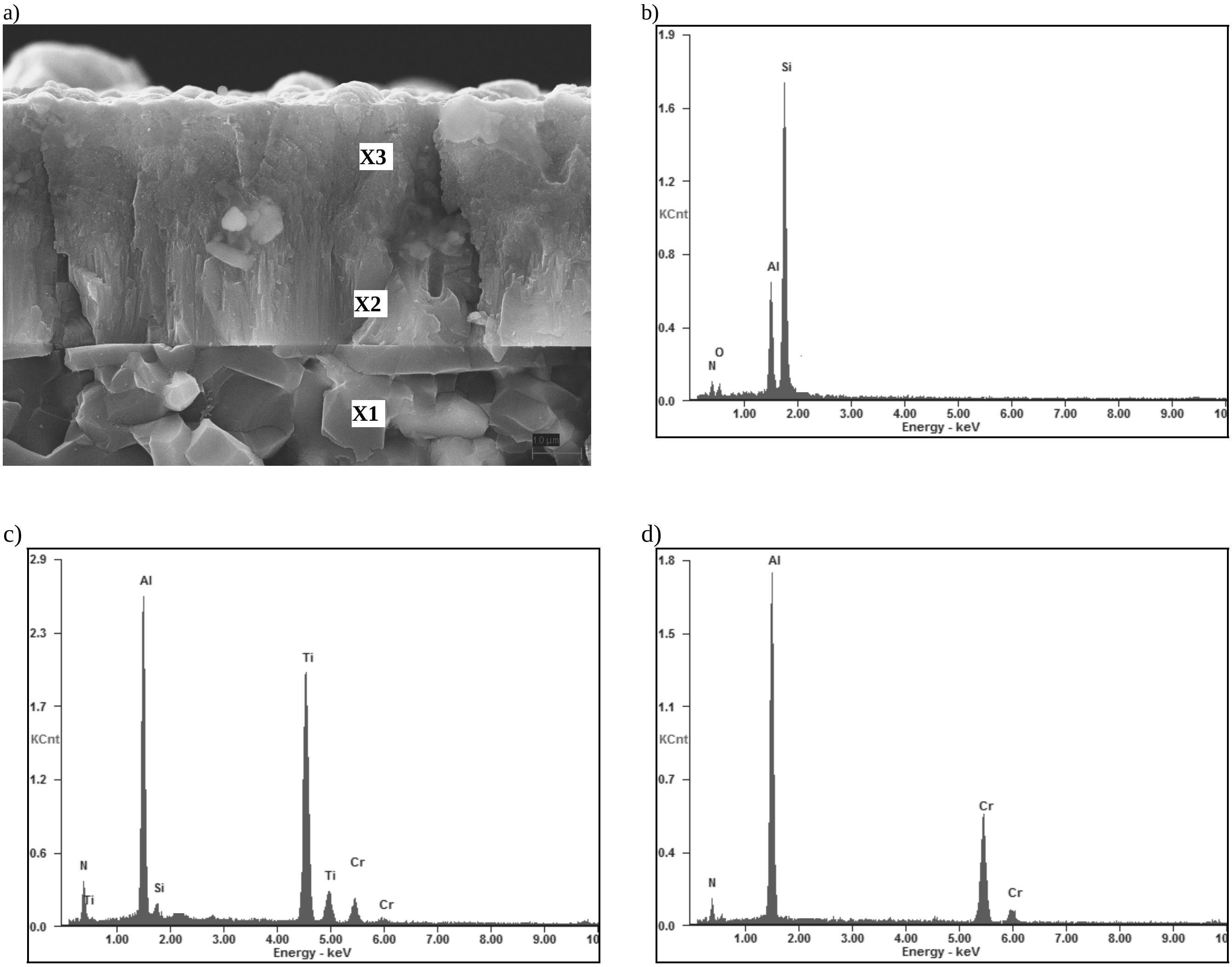


Fig. 1. Fracture of the (Ti,Al)N+(Al,Cr)N coating deposited onto the sialon ceramics substrate and X-ray energy dispersive plots the area as in a coating b) region X1, c) region X2, d) region X3

In effect of the qualitative X-ray microanalysis we obtained information about the elements present in the selected microareas of the investigated coatings (Fig. 1), and in effect of the quantitative analysis we obtained information about mass and atomic concentration of particular elements (Tables 2, 3, 4). The qualitative and quantitative analysis EDS from the microareas of the coating demonstrates that the investigated layers contain elements appropriate for a given coating.

Table 2.
Pointwise chemical composition analysis from substrate SiAlON (Fig. 1b)

Element	The mass concentration of main elements, Weight, %	The mass concentration of main elements, Atomic, %
N	16.06	26.66
O	05.26	07.65
Al	16.62	14.32
Si	62.06	51.37

Table 3.
Pointwise chemical composition analysis from (Ti,Al)N layer (Fig. 1c)

Element	The mass concentration of main elements, Weight, %	The mass concentration of main elements, Atomic, %
N	12.34	29.60
Al	25.53	31.78
Si	01.38	01.65
Ti	44.83	31.43
Cr	05.95	03.84

Table 4.
Pointwise chemical composition analysis from (Al,Cr)N layer (Fig. 1d)

Element	The mass concentration of main elements, Weight, %	The mass concentration of main elements, Atomic, %
N	13.06	27.50
Al	44.03	48.15
Cr	42.91	24.35

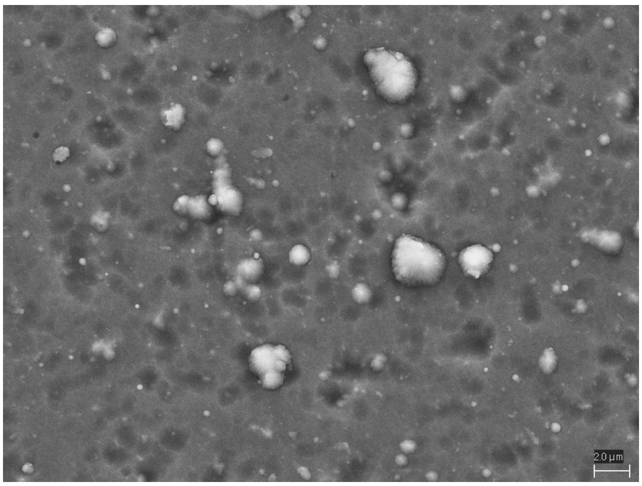


Fig. 2 Surface topography of the (Ti,Al)N+(Al,Cr)N coating

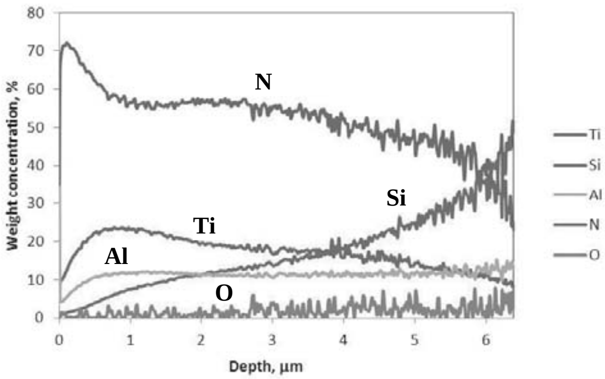
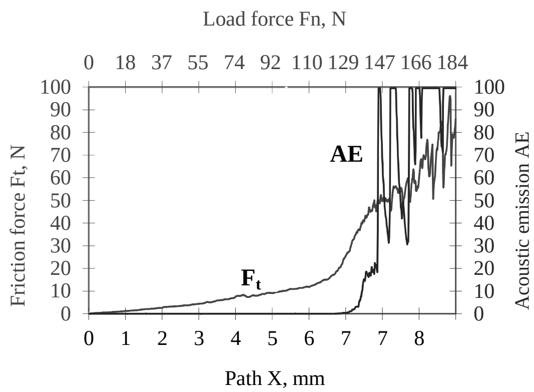


Fig. 3. Changes of concentrations of constituents of the (Ti,Al)N coating and of the substrate materials sialon tool ceramics analysed in GDOS spectrometer

a)



b)

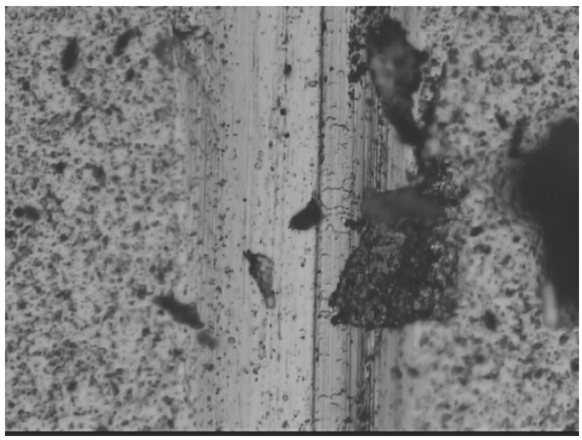


Fig. 4. a) Acoustic emission (AE) and friction force F_t as a function of the load force F_n for (Ti,Al)N+(Al,Cr)N coating on sialon tool ceramics; b) scratch failure at L_c = 123 N, (mag. 200x)

Examinations in the glow discharge optical emission spectrometer GDOS make it possible only to evaluate the qualitative differences of the chemical compositions in the selected micro-area of each specimen. Basing on these examinations, certain regularity was found out of the distribution of elements included in both coatings and substrate. The analysis made using the glow discharge optical emission spectrometer GDOS indicates that in the analysed cases, in the joint zone, concentration of elements included in the substrate grows from the coating surface with the simultaneous decreasing concentration of elements constituting the coatings (Fig. 3).

In the consequence of hardness measurements investigated uncoated samples and coated with the PVD coatings it was found a increase surface microhardness after deposited coatings (Table 1). It was also found that a (Ti,Al)N+(Al,Cr)N coating is shown higher hardness than (Al,Cr)N and (Al,Cr)N+(Ti,Al)N coatings.

The characteristic critical load L_c was determined by scratch test method. During scratch testing the friction force F_t and acoustic emission AE were registered as a function of the load F_n. The value of the critical load L_c is a point on the curve of the friction force at which first damage of the coating was observed and corresponding acoustics emission signal was registered (Fig. 4a). Scratch adhesion tracks were analysed using the light microscope coupled with a measuring gauge. Thus the values of the critical load L_C could be obtained on the basis of metallographic observations (Fig. 4b).

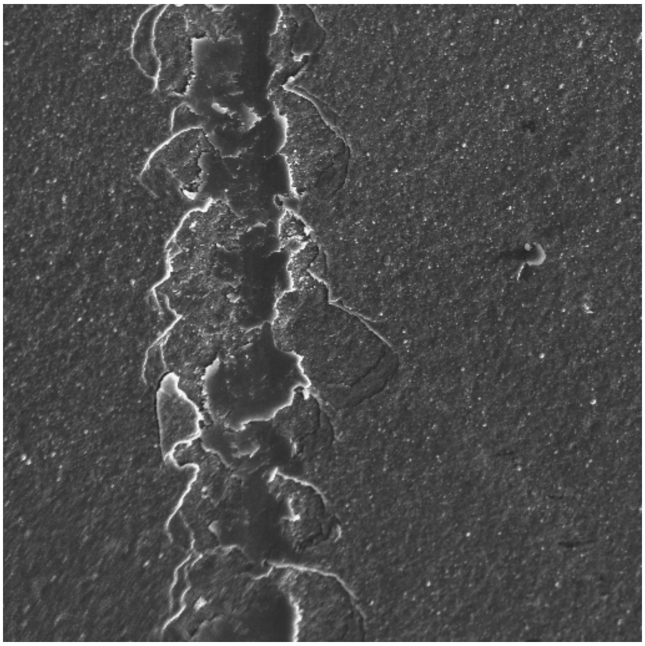


Fig. 5. Failure characteristic obtained by scratch test of the (Al,Cr)N+(Ti,Al)N coating deposited on sialon tool ceramics

The results of the scratch test measurement for the studied coatings are shown in Table 1. It was fund that (Ti,Al)N+(Al,Cr)N coating show very good adherence to substrate L_c = 123 N. However, the (Ti,Al)N coating adherence to sialon substrate is low. Observations in the scanning electron microscope of scratches arose as result of the Scratch Test show that a dominant mechanism of failure coatings with very good adherence was both a abrasion and cohesion cracking. In a case of coating indicate low adherence, it was found that both a delamination and an extensive cracks as well as crushing.

It was found out during adhesion tests of coatings deposited onto the sialon tool ceramics substrates that spalling and delamination are the most common forms of their defects. The (Ti,Al)N+(Al,Cr)N coating revealed only single damages in the form of delamination on the edge and inside of the scratch after applying the load. These damages were getting more concentrated on the scratch surface along with increasing the load and one could observe partial and band delaminations, as well as single side and two-side spalling (Fig. 5).

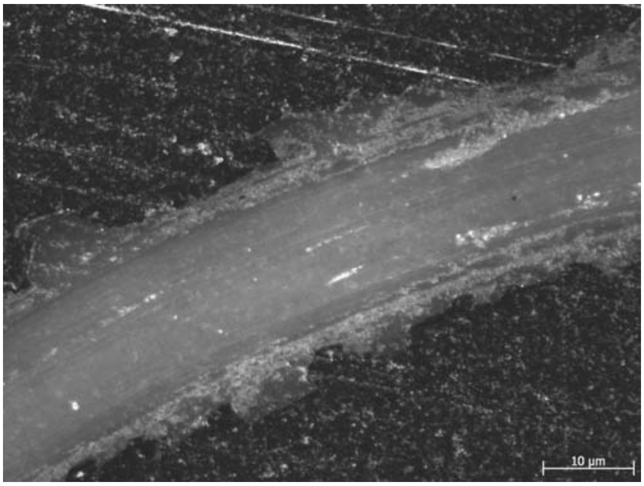


Fig. 6. Trace of the tribological failure on the surface of (Ti,Al)N coating put down deposited on sialon tool ceramics

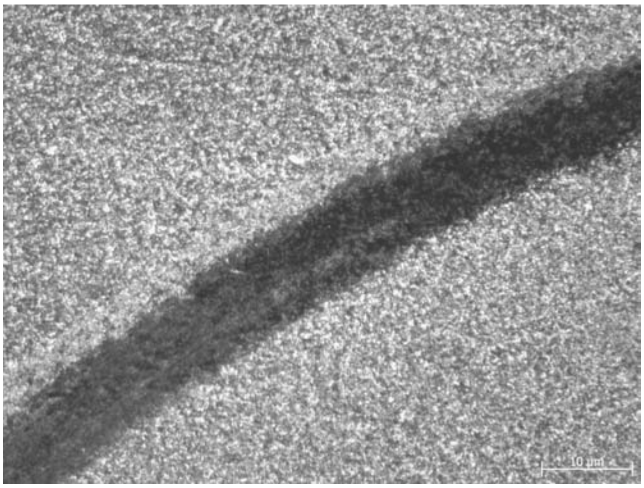


Fig. 7. Trace of the tribological failure on the surface of (Al,Cr)N coating put down deposited on sialon tool ceramics

Fig. 8. Trace of the tribological failure on the surface of (Al,Cr)N+(Ti,Al)N coating deposited on sialon tool ceramics

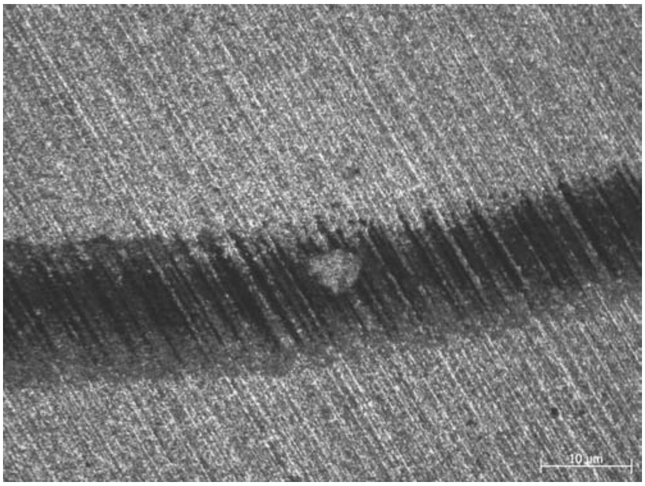


Fig. 9. Trace of the tribological failure on the surface of (Ti,Al)N+(Al,Cr)N coating put down deposited on sialon tool ceramics

It was found out basing on the abrasion wear resistance of coatings deposited with the PVD technology that all coatings of this group display good tribological properties. The (Ti,Al)N coating is characteristic of the extensive adhesion damages both in the tribological ridge and outside of it (Fig. 6). Next, the (Al,Cr)N and (Al,Cr)N+(Ti,Al)N coatings display minute locations in the tribological ridge where coating damage has reached the substrate material (Figs. 7, 8). No damages reaching the substrate material were observed in case of the (Ti,Al)N+(Al,Cr)N coating (Fig. 9). For all the investigated PVD coatings adherence occurs of the damaged coating and of the employed counter-specimen material, which directly affects the variable friction coefficient values. Coefficient of friction ranges from 0.4 to 0.6 for the two-layer (Al,Cr)N+(Ti,Al)N and (Ti,Al)N+(Al,Cr)N coatings; however, the friction coefficient reaches values from 0.7 for other (Ti,Al)N coating.

5. Summary

The compact structure of the coatings without any visible delamination was revealed on the scanning electron microscope. The investigated coatings show columnar structure which may be identified as that characteristic for the zone IV of the Thornton model. Numerous microdroplets characteristic for the CAE method of deposition are presented in the coatings

The investigations of the adhesion carried out using of the scratch test revealed cohesive and adhesive properties of the coatings. It has been found, on the basis of these examinations, that the value of critical load L_C is between 21 N and 123 N. The greatest value of the critical load has been obtained for the (Ti,Al)N+(Al,Cr)N coating. Probably, the good adhesion of these coatings to sialon substrate is connected with the same type of bonding in coat and sialon substrate. The good properties of the PVD multicomponent coatings make them suitable for various engineering and industrial applications.

All the same, the investigated PVD coatings have the relatively good tribological properties, which are proven also by a high number of load cycles – up to 10,000. The (Ti,Al)N+(Al,Cr)N coating demonstrates a high wear resistance with this static load method. The failures of this coating type do not reach the substrate material after loading with 10,000 cycles, what can be an effect of very high microhardness value of coating (=3120 HV0.5). It is very difficult during the tribological analysis to assess the dimensions of the failure, especially in case of coatings whose failures do not reach the substrate material. During observations made using the light microscope in the tribological failure trace the particles of the damaged coating or of the counter-specimen are revealed as defects reaching down to the substrate material, which results in stopping the analysis too early. Generally, the PVD coatings, and most of all the (Ti,Al)N+(Al,Cr)N (Al,Cr)N and (Al,Cr)N+(Ti,Al)N ones may be regarded as the most wear resistant coatings.

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References

[1] A.E. Reiter, V.H. Derflinger, B. Hanselmann, T. Bachmann, B. Sartory, Investigation of the properties of Al1-xCrxN coatings prepared by cathodic arc evaporation, Surface & Coatings Technology 200 (2005) 2114-2122.

[2] B.A. Movchan, G.S. Marinski, Gradient protective coatings of different applications produced by EB-PVD, Surface and Coatings Technology 100-101 (1998) 309-315.

[3] B.A. Movchan, K.Yu Yakovchuk, Graded thermal barrier coatings, deposited by EB-PVD, Surface and Coatings Technology 188-189 (2004) 85-92.

[4] D. Pakuła, L.A. Dobrzański, A. Križ, M. Staszuk, Investigation of PVD coatings deposited on the Si_3N_4 and sialon tool ceramics, Archives of Materials Science and Engineering 46/1 (2010) 53-60.
[5] D. Pakuła, L.A. Dobrzański, K. Gołombek, M. Pancielejko, A. Křiž, Structure and properties of the Si_3N_4 nitride ceramics with hard wear resistant coatings, Journal of Materials Processing Technology 157-158 (2004) 388-393.
[6] L.A. Dobrzański, D. Pakuła, E. Hajduczek, Structure and properties of the multi-component TiAlSiN coatings obtained in the PVD process in the nitride tool ceramics, Journal of Materials Processing Technology 157-158 (2004) 331-340.
[7] K. Lukaszkowicz, J. Sondor, A. Kriz, M. Pancielejko, Structure, mechanical properties and corrosion resistance of nanocomposite coatings deposited by PVD technology onto the X6CrNiMoTi17-12-2 and X40CrMoV5-1 steel substrates, Journal of Materials Science 45 (2010) 1629-1637.
[8] K. Lukaszkowicz, L.A. Dobrzański, A. Zarychta, Structure, chemical and phase compositions of coatings deposited by reactive magnetron sputtering onto the brass substrate, Journal of Materials Processing Technology 157-158 (2004) 380-387.
[9] L.A. Dobrzański, D. Pakuła, A. Křiž, M. Soković, J. Kopač, Tribological properties of the PVD and CVD coatings deposited onto the nitride tool ceramics, Journal of Materials Processing Technology 175 (2006) 179-185.
[10] L.A. Dobrzański, D. Pakuła, Comparison of the structure and properties of the PVD and CVD coatings deposited on nitride tool ceramics, Journal of Materials Processing Technology 164-165 (2005) 832-842.
[11] L.A. Dobrzański, D. Pakuła, Structure and properties of the wear resistant coatings obtained in the PVD and CVD processes on tool ceramics, Materials Science Forum 513 (2006) 119-133.
[12] L.A. Dobrzański, J. Mikuła, K. Gołombek, Structural characteristics of the modern sintered tool materials, Materials Science Forum 530-531 (2006) 499-504.
[13] L.A. Dobrzański, J. Mikuła, The structure and functional properties of PVD and CVD coated $Al_2O_3 + ZrO_2$ oxide tool ceramics, Journal of Materials Processing Technology 167/2-3 (2005) 438-446.
[14] L.A. Dobrzański, K. Gołombek, E. Hajduczek, Structure of the nanocrystalline coatings obtained on the CAE process on the sintered tool materials, Journal of Materials Processing Technology 175 (2006) 157-162.
[15] L.A. Dobrzański, K. Gołombek, J. Kopač, M. Soković, Effect of depositing the hard surface coatings on properties of the selected cemented carbides and tool cermets, Journal of Materials Processing Technology 157-158 (2004) 304-311.
[16] L.A. Dobrzański, K. Gołombek, Characterisation of the gradient coatings TiN+(Ti,Al,Si)N+TiN type deposited on sintered tool materials, La Metallurgia Italiana 4 (2006) 29-34.
[17] L.A. Dobrzański, K. Lukaszkowicz, A. Kriz, Properties of the multi-layer Ti/CrN and Ti/TiAlN coatings deposited with the PVD technique onto the brass substrate, Journal of Materials Processing Technology 143-144 (2003) 832-837.
[18] L.A. Dobrzański, L.W. Żukowska, J. Mikuła, K. Gołombek, T. Gawarecki, Hard gradient (Ti,Al,Si)N coating deposited on composite tool materials Archives of Materials Science and Engineering 36/2 (2009) 69-75.
[19] L.A. Dobrzański, M. Staszuk, K. Gołombek, A. Śliwa, M. Pancielejko, Structure and properties PVD and CVD coatings deposited onto edges of sintered cutting tools, Archives of Metallurgy and Materials 55/1 (2010) 187-193.
[20] L.A. Dobrzański, M. Staszuk, PVD and CVD gradient coatings on sintered carbides and sialon tool ceramics, Journal of Achievements in Materials and Manufacturing Engineering 43/2 (2010) 552-576.
[21] M. Betiuk, T. Borowski, K. Burdyński, The (Ti,Al)N, (Ti,Al)C and (Ti,Al)CN multicompound coatings synthesis in low pressure of DC arc discharge, Engineering Materials 6 (2008) 674-678 (in Polish).
[22] M. Cłapa, D. Batory, Improving adhesion and wear resistance of carbon coatings using Ti:C gradient layers, Journal of Achievements in Materials and Manufacturing Engineering 20 (2007) 415-418.
[23] M. Soković, J. Kopač, L.A. Dobrzański, J. Mikuła, K. Gołombek, D. Pakuła, Cutting characteristics of PVD and CVD - Coated ceramic tool inserts, Tribology in Industry 28/1-2 (2006) 3-8.
[24] M. Staszuk, The structure and properties of PVD and CVD gradient coatings deposited on sialons and sintered carbides, PhD Thesis, Silesian University of Technology Library, Gliwice, 2009 (in Polish).
[25] M. Wysiecki, Contemporary Tool Materials, WNT, Warsaw, 1997 (in Polish).
[26] S. PalDey, S.C. Deevi, Properties of single layer and gradient (Ti,Al)N coatings, Materials Science and Engineering A 361 (2003) 1-8.
[27] Shuichi Kawno, Junichi Takahashi, Shiro Shimada, The preparation and spark plasma sintering of silicon nitride-based materials coated with nano-sized TiN, Journal of the European Ceramic Society 24 (2004) 309-312.
[28] T. Liu, C. Dong, S. Wu, K. Tang, J. Wang, J. Jia, TiN gradient and Ti/TiN multi-layer protective coatings on Uranium, Surface and Coating Technology 201 (2007) 6737-6741.
[29] T. Wierzchoń, Structure and properties of multicomponent and composite layers produced by combined surface engineering methods, Surface and Coatings Technology 180-181 (2004) 458-464.
[30] Yin-Yu Chang, Da-Yung Wang, Chi-Yung Hung, Structural and mechanical properties of nanolayered TiAlN/CrN coatings synthesized by a cathodic arc deposition process, Surface and Coatings Technology 200 (2005) 1702-1708.
[31] L. Cunha, L. Rebouta, F. Vaz, M. Staszuk, S. Malara, J. Barbosa, P. Carvalho, (...), J.P. Riviere, Effect of thermal treatments on the structure of MoNxOy thin films, Vacuum 82/12 (2008) 1428-1432.
[32] L.A. Dobrzański, M. Staszuk, J. Konieczny, W. Kwaśny, M. Pawlyta, Structure of TiBN coatings deposited onto cemented carbides and sialon tool ceramics Archives of Materials Science and Engineering 38/1 (2009) 48-54.
[33] L.A. Dobrzański, D. Pakuła, J. Mikuła, K. Gołombek, Investigation of the structure and properties of coatings deposited on ceramic tool materials, International Journal of Surface Science and Engineering 1/1 (2007) 111-124.