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Surface modification of sialon ceramics and cemented carbides by PVD coating deposition

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Materials

ABSTRACT

Purpose: The paper includes investigation results of structures and mechanical properties of coatings deposited by the physical vapor deposition (PVD) techniques onto both sialon tool ceramics and sintered carbides. The paper includes two kinds of coating materials, isomorphic containg phases with TiN and AlN.
Design/methodology/approach: In the paper were presented some observations of coating structures, before carried out in the scanning electron microscope. Phases composition analysis was carried out with using a XRD and GIXRD method. The roughness of surface measurements, microhardness tests and adhesion coatings to substrates tests were carried out It was found that some coatings showed a fine-grained structure. Cutting ability were defined on basis of technological cutting trials.
Findings: Coatings, which had contained a AlN phase about hexagonal lattice showed a considerably higher adhesion to substrate from sialon ceramics rather than a coating contained a TiN phase about cubic lattice. As a result of setting coatings onto substrates, it was found a significant increase of both coatings hardness and surface roughness. The coatings contains a TiN phase shows a low adherence to substrate ceramic, what is the consequence of low cutting ability. Whereas, a coatings contains a AlN phase about hexagonal lattice show very good adherence to ceramic substrate as well as very good cutting ability.
Research limitations/implications: The good adhesion (Al,Ti)N coating to sialon substrate is connecting with the same type of bonding in coat and sialon substrate.
Originality/value: The good properties of the PVD gradient coatings make them suitable for various engineering and industrial applications.
Keywords: Tool materials; Surface treatment; Thin and thick coatings; Wear resistance

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1. Introduction

Development of surface engineering is an exactly connected with a fact that utilitarian properties of many products depend on in significant grade from a structure and properties of surface layers, but not only from possibilities to carry the mechanical loads by all active section element [1-15]. Therefore, in area of the surface engineering are search some new solutions driving at bettering resistance of tool material on the abrasive wear and dynamic machining. The improvement of utilitarian properties of tools and decrease ecological dangers take place as a result of applying hard coatings obtained in the PVD and CVD processes, mainly as a result of tribological contact bettering conditions in the machining area and elimination of machining liquid [1-14].

The classification of protective coatings for the sake of a type of atomic bond dominated in a given coating type is presented in Fig. 1. The materials with the predominant number of metallic bonds are presented the most numerous group among all types of coating materials. Nitrides and carbides of transition metals, as well some borides and silicides belong to this group. The metallic-covalence bonds are presented in a large minority of these phases, that’s why these materials connected to themselves high hardness and abrasive resistance with the resistance to brittle cracking, so higher than phases of covalence and ionic bonds. Materials with the prevalence of ionic groups is an another group of coating materials. Mainly oxides belong to this group. Materials with the prevalence of covalence bonds are presented the third group, we belong to it diamond coatings and those made from boron nitride. The highest hardness is pointed out by this group of materials [1,15-20].

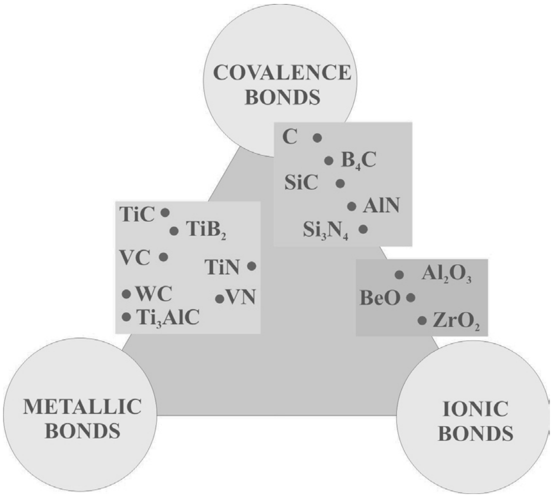

Fig. 1. Dependence of hard material coatings on a type of bond [16,17,18]

The main objective of this research was an investigation of both the coatings structure and mechanical properties deposited by the physical vapor deposition (CAE-PVD) on sintered carbides and sialon tool ceramics substrates.

2. Materials

The research was carried out on cutting edges made from sintered carbides of WC-Co type and sialon tool ceramics, uncoated and coated with PVD coatings. The substrates were ultrasonic cleaned in pure acetone and exsiccated in dry heat stream. The process of coatings deposition was made with use of device based on the cathodic arc evaporation (CAE) method. The following coatings were investigated such as: (Ti,Al)N, (Al,Ti)N, (Al,Cr)N and Ti(C,N) gradient coatings.

3. Research methodology

In the scanning electron microscope Supra 35 of Zeiss Company was observed the surface topography and the structure of the produced coatings along the transverse fractures. The detection of secondary electrons (SE) and backscattered electrons (BSE) with the accelerating voltage within the range of 5-20 kV was applied to obtain the images of the investigated samples.

The method used to carry out the qualitative and quantitative analysis of the chemical composition in the micro-areas of the investigated coatings was the X-ray energy dispersive spectroscopy (EDS) with the application of the spectrometer EDS LINK ISIS of the Oxford Company, being a component of the electron scanning microscope Zeiss Supra 35. The researches were carried out with the accelerating voltage of 20 kV.

Based on investigations in the glow discharge optical spectrometer GDOS-750 QDP of the Leco Instruments Company the following changes were determined: chemical concentration of the coating components along the direction perpendicular to its surface, and the concentration in the transit zone between the coating and the substrate material. The following operating conditions of the Grimm tube of the spectrometer were applied: operating pressure 100 Pa, voltage feed to the tube 700 V, tube current 20 mA, internal diameter of the tube 4 mm.

In the transmission electron microscope JEM 3010 UHL of the JEOL Company, with the accelerating voltage of 300kV and maximum magnification of 25000 times were carried out the observation of the structure of thin foils and the diffraction researches. The computer program called “Eldyf” was applied to solve the diffractograms from the transmission electron microscope.

The method used to analyze the phase composition of the substrates and the obtained coatings was the X-ray diffraction (XRD) on the X-ray apparatus X’Pert of the Panalytical Company using the filtered radiation of a cobalt lamp. In further experimental investigations was applied the grazing incident X-ray diffraction technique (GIXRD) in order to obtain more accurate information from the surface layer of the investigated materials, because of the superposition of reflexes of the substrate material and coating and due to their intensity hindering the analysis of the obtained results.

Based on X-ray diffraction patterns obtained by the grazing incident X-ray diffraction technique (GIXRD) using the Scherrer’s method was evaluated the grain size in the investigated coatings.

There were carried out on the profilographometer Surtronic 3+ of Taylor Hobson Company the measurements of roughness of the polished samples surface from sintered carbides of the WC-Co type and of sialon ceramics without coating and covered with the investigated coatings, while the roughness measurement of the surface of gray cast iron, after the technological machining trial with cutting edges without coatings and with the investigated coatings was performed on the profilographometer Diavite Compact of Asmeo Ag Company. The measurement length of Lc=0.8 mm and measurement accuracy of ± 0.02 µm was assumed. The quantity describing the roughness was assumed as a parameter Ra acc. the Standard PN-EN ISO 4287:1999. During investigations were performed six measurements on each of the investigated samples and determined the following statistical parameters average, standard deviation and confidence interval, assuming the confidence factor at 1-α=0.95.

The Vickers method was applied to determine the hardness of the investigated materials. The classical Vickers method in the mode 'load-unload', using the loading equal to 3N according to the Standard PN-EN ISO 6507-1:2007 was applied to investigate the hardness of the produced coatings, that is the hardness of the covered substrates from sintered carbides and sialon tooling ceramics.

Based on the Scratch Test analysis on the apparatus Revetest of the CSEM Company was determined the adhesion of the coatings to the substrate. The critical load Lc at which the adhesion of the coating fails was determined basing on the value of acoustic emission recorded during the measurement and on the observation of scratches formed during the scratch test. In the light microscope being a component of the apparatus were made these observations. Whereas in the scanning electron microscope DSM-940 of the Opton Company, with the accelerating voltage of 20kV were made much more detailed observations of the formed damages.

Basing on cutting trials without cutting tool lubricants on the lathe TUR 630M were carried out the tests involving the cutting ability of inserts from sintered carbides and sialon ceramics covered and non-covered with PVD and CVD coatings. The gray cast iron EN-GJL-250 of the hardness about 215 HB was applied to the machining at room temperature. There was determined basing on the measurements of the width of wear band on the tool flank the durability of the investigated inserts. The measurement of both the average width of flank wear VB and of the maximum width of wear band VB_{max} was performed using the light microscope Carl Zeiss Jena. When the assumed wear criterion for after-machining of VB=0.2 mm was exceeded then the machining trials were being stopped. The scanning electron microscope Zeiss Supra 35 was applied to make observation of the wear of tool flank and attack surface of the machining inserts. The analysis of chemical composition in the micro-areas was carried out using the EDS method. In a graphical form as the relation of wear band on the tool flank VB in the function of cutting trial time was presented the obtained results. The durability of the cutting edge is defined as the time T [min], after which the value of the assumed criterion VB=0.2 mm is exceeded.

4. Experimental results

It was found the presence of an isomorphic phases from TiN using the X-ray diffraction patterns obtained from the (Ti,Al)N coating. In a case of the Ti(C,N) coating, it was confirmed the presence of titanium carbo-nitride, however in a case of the (Al,Ti)N and (Al,Cr)N coatings the diffraction analysis confirmed the presence of A1N phase of the hexagonal lattice in both coatings and of phases respectively TiN and CrN. Results of these investigations were presented in the paper [1].

The investigations of thin foils from (Al,Ti)N coating deposited on the substrate from sintered carbides and sialon tooling ceramics confirm that in congruence with the assumptions the produced coatings contain of AlN phase of the hexagonal lattice (spatial group P63mc) and TiN.

In the scanning electron microscope were carried out the fractographic tests, based on them it was found that the PVD coatings are uniformly deposited and closely adhere to the substrate. From the fractures of (Al,Cr)N coatings, it can be observed that this coating is also laminar, typical for multi-component coatings obtained through the application of separate sources of metal pairs Cr and Al (Fig. 2). Based on the observation of the fractures of PVD coatings that the coatings (Al,Ti), Ti(C,N) and (Al,Cr)N deposited on both substrates (Fig. 3), it was found that they have a structure classified into zone T according to the Thornton model, as well as the coating (Ti,Al)N deposited on sintered carbides. However, the coating (Ti,Al)N produced on sialon inserts has the structure of thicker column grains (zone II acc. Thornton model).

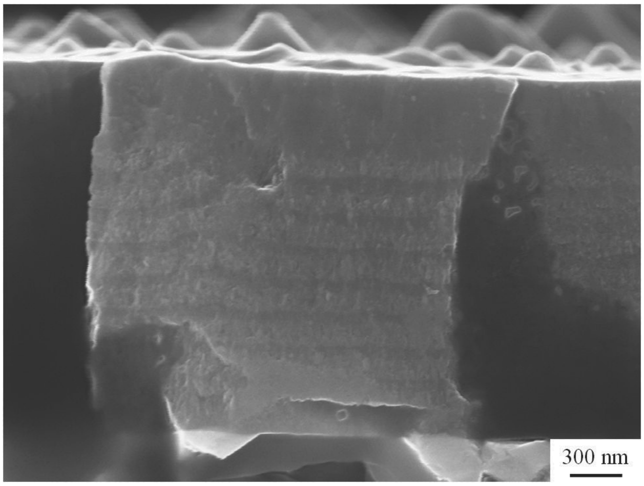

Fig. 2. Fracture of the (Al,Cr)N coating deposited onto the sintered carbides substrate

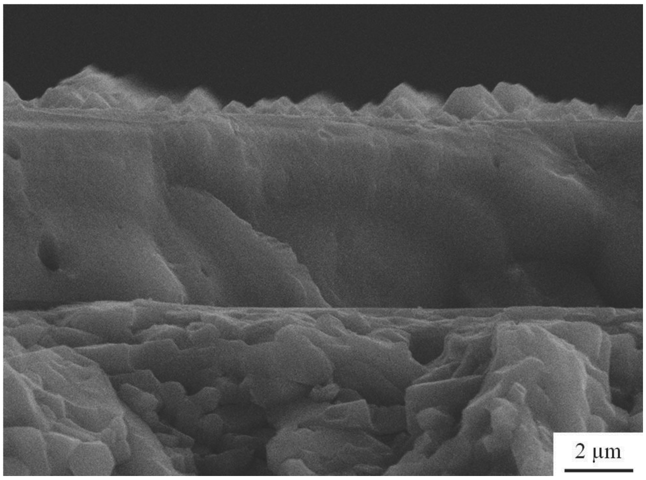

Fig. 3. Fracture of the (Al,Ti)N coating deposited onto the sialon ceramics substrate

As a results of the metallographic tests performed on the scanning electron microscope, it was found that the surface morphology of coatings produced with the PVD technique on sintered carbides of the WC-Co type and on tooling sialon ceramics is characterized by high non-homogeneity connected with the presence of numerous droplet-shaped microparticles (Figs. 4, 5). The presence of these morphological defects is connected with the nature of cathode arc evaporation.

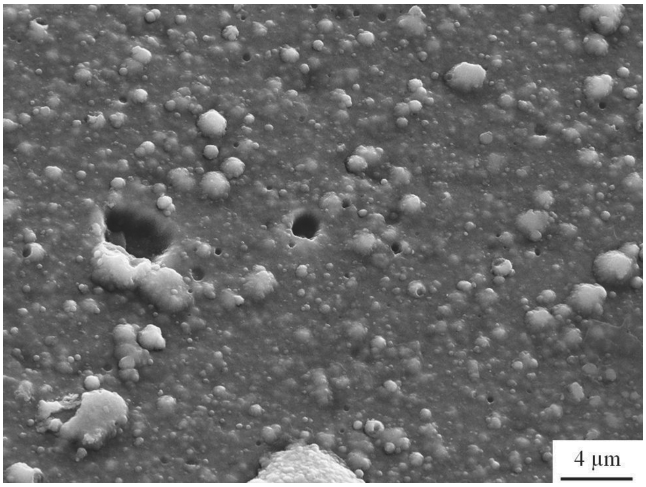

Fig. 4. Surface topography of the (Al,Cr)N coating deposited onto the sialon ceramics substrate

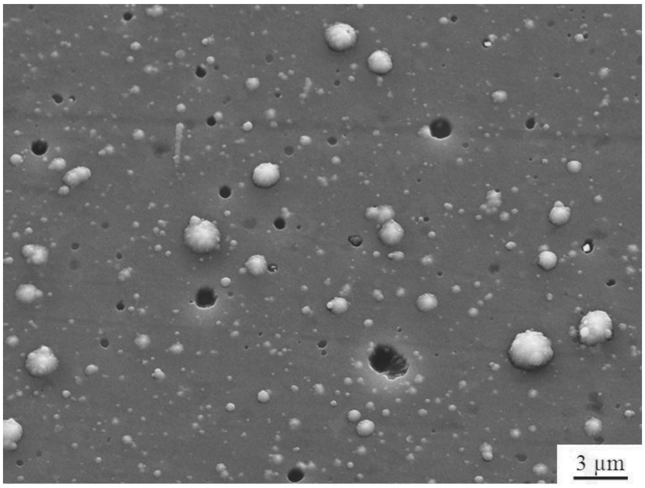

Fig. 5. Surface topography of the (Al,Cr)N coating deposited onto the sintered carbides substrate

It was found that the morphology of coating surfaces (Fig. 6) has an influence on the rise of roughness Ra of the surfaces of inserts from sintered carbides and sialon ceramics covered by the researched coatings (Table 1).

The existence of the transition zone between substrate material and coating, improving adhesion of the deposited coatings to the substrate was confirmed by the GDOES tests. In the transition zone the concentration of elements included in the substrate grows, while the concentration of elements constituting the coatings decreases rapidly. Its development may be also connected with high energy ions causing transfer of the elements in the connection zone, increase of desorption of the substrate surface, and development of defects in the substrate. It was found that the results obtained with the use of the GDOES cannot be interpreted unequivocally, due to the inhomogeneous vaporization of the specimen material during the tests. The changes of the chemical concentrations of the coating constituents and substrate material upon tests performed on the glow-discharge optical emission spectroscope are presented in Fig. 7.

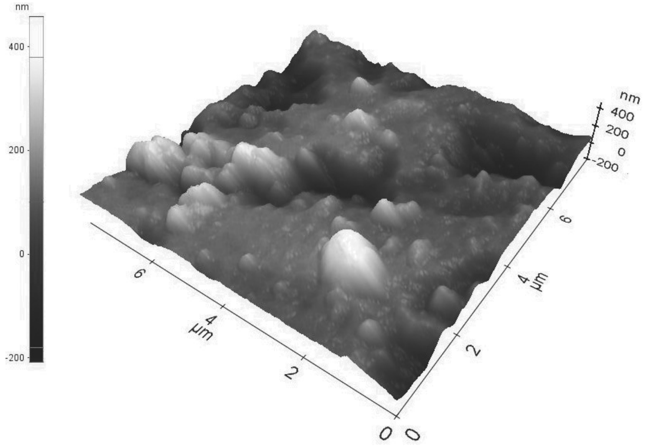

Fig. 6. Surface topography of the (Al,Cr)N coating (AFM)

As a result of microhardness investigations of sintered carbides and sialon ceramics, uncoated and coated investigated coatings, it was found that microhardness increase of surface layer cutting edges after deposited investigating coatings (Table 2).

The coating-substrate adherence depends on both the chemical and phase composition of coatings (Figs. 8, 9, Table 2). This relation is particularly relevant with respect to PVD coatings on the substrate performed from sialon ceramics. The coatings contain only TiN and Ti(C,N) phases have low adhesion to the sialon substrate - Lc=13÷36 N, the coatings contain the AlN phases are characterized by very good adhesion to the substrate - Lc=53÷112 N. It is worth to remember that sialons belong to covalence ceramics, so in the coatings contain isomorphic phases with titanium nitride TiN are metallic bonds, which results in low adhesion of these coatings to the substrate of different bond. In the case of coatings contain AlN phase of the hexagonal lattice there were found the covalence bonds analogous to the ceramic substrate, which yields good adhesion of these coatings to the substrate. It was found that a type of interatomic bonds presented in the material of the substrate and coating has a great influence on the coating-substrate adherence. The adhesion of the coating to the substrate performed from sintered carbides is conditioned among others, apart from adhesion, by a slight diffusive displacement of elements in the contact zone effected by the implantation of high energy ions falling down on the negatively polarized substrates.

Table 1.
Roughness of investigated samples and thickness of investigated coatings

Substrate	Coating	Roughness R_a, µm					Thickness, µm
		min. value	max. value	mean value	Standard deviation	Confidence interval for α=0.05	
Sintered carbides	Uncoated	0.06	0.06	0.06	0	±0	-
	Ti(C,N)	0.38	0.68	0.50	0.12	±0.12	2.1
	(Ti,Al)N	0.24	0.78	0.39	0.20	±0.21	3.5
	(Al,Ti)N	0.14	0.26	0.18	0.04	±0.05	2.5
	(Al,Cr)N	0.16	0.36	0.26	0.07	±0.07	3.8
Sialon ceramics	Uncoated	0.06	0.06	0.06	0	±0	-
	Ti(C,N)	0.34	0.46	0.38	0.05	±0.05	1.8
	(Ti,Al)N	0.24	0.32	0.28	0.03	±0.04	5.0
	(Al,Ti)N	0.14	0.18	0.15	0.02	±0.02	3.0
	(Al,Cr)N	0.24	0.36	0.31	0.05	±0.05	4.8

Table 2.
Microhardness of investigated samples and critical load of investigated coatings

Substrate	Coating	Microhardness, HV					Critical load L_c, N
		min. value	max. value	mean value	Standard deviation	Confidence interval for α=0.05	
Sintered carbides	Uncoated	1789	1865	1826	26	±21	-
	Ti(C,N)	2802	3551	3101	278	±219	77
	(Ti,Al)N	2757	3822	3327	494	±401	109
	(Al,Ti)N	2802	3861	3301	369	±300	100
	(Al,Cr)N	2348	3534	2867	501	±408	96
Sialon ceramics	Uncoated	1990	2080	2035	31	±25	-
	Ti(C,N)	2599	3100	2842	183	±149	26
	(Ti,Al)N	2545	3259	2961	249	±203	21
	(Al,Ti)N	3117	3884	3600	314	±255	112
	(Al,Cr)N	1845	2962	2229	406	±330	53

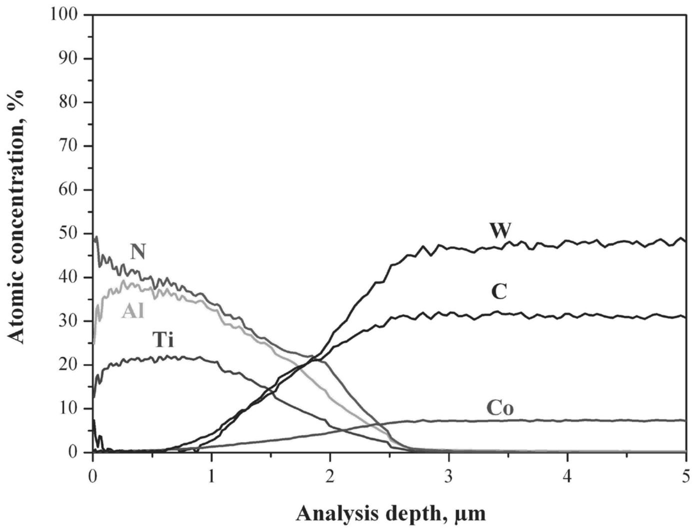

Fig. 7. Changes of constituent concentration of the (Al,Ti)N coating and the sintered carbides substrate material

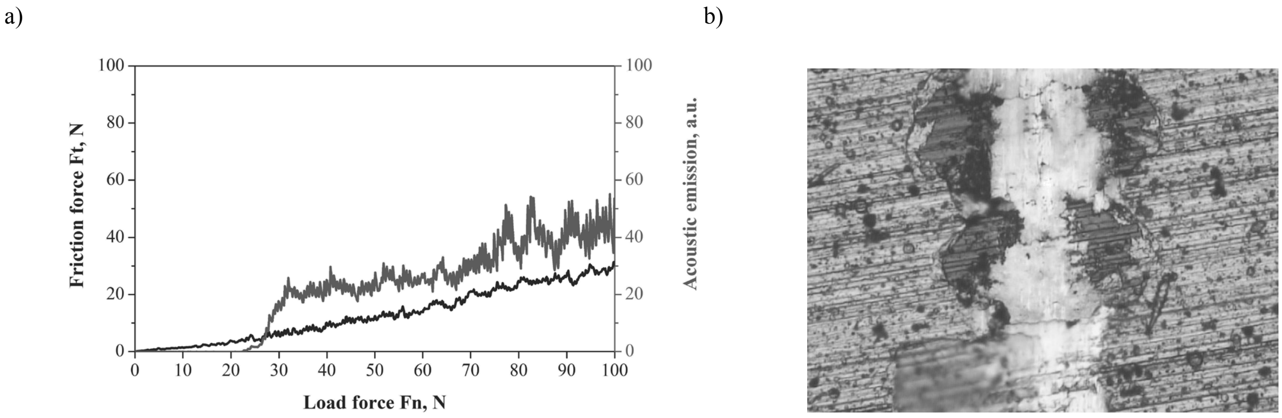

Fig. 8. a) Acoustic emission (AE) and friction force Ft as a function of the load Fn for Ti(C,N) coating on sialon ceramics substrate, b) scratch failure at Lc (opt) = 27 N, mag. 200 x

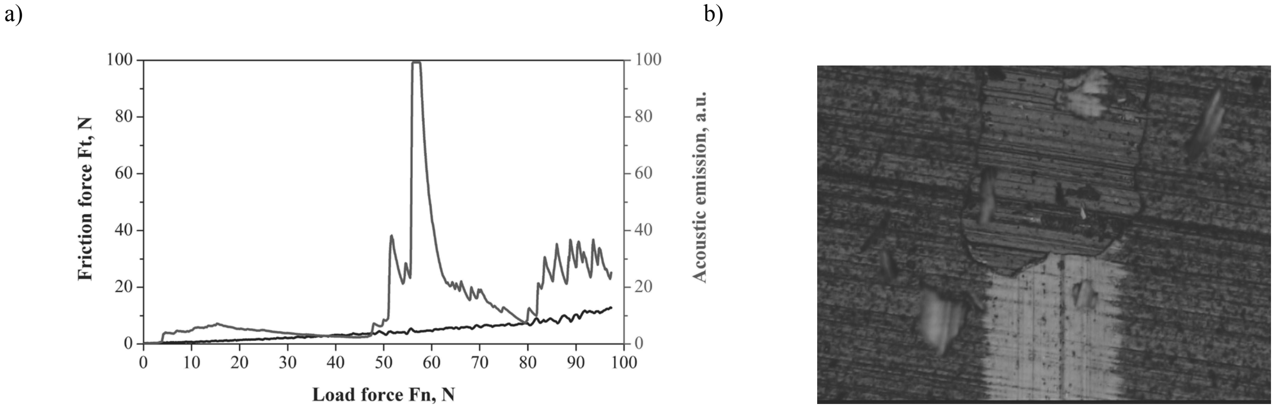

Fig. 9. a) Acoustic emission (AE) and friction force Ft as a function of the load Fn for (Al,Cr)N coating on sialon ceramics substrate, b) scratch failure at Lc (opt) = 53 N, mag. 200 x

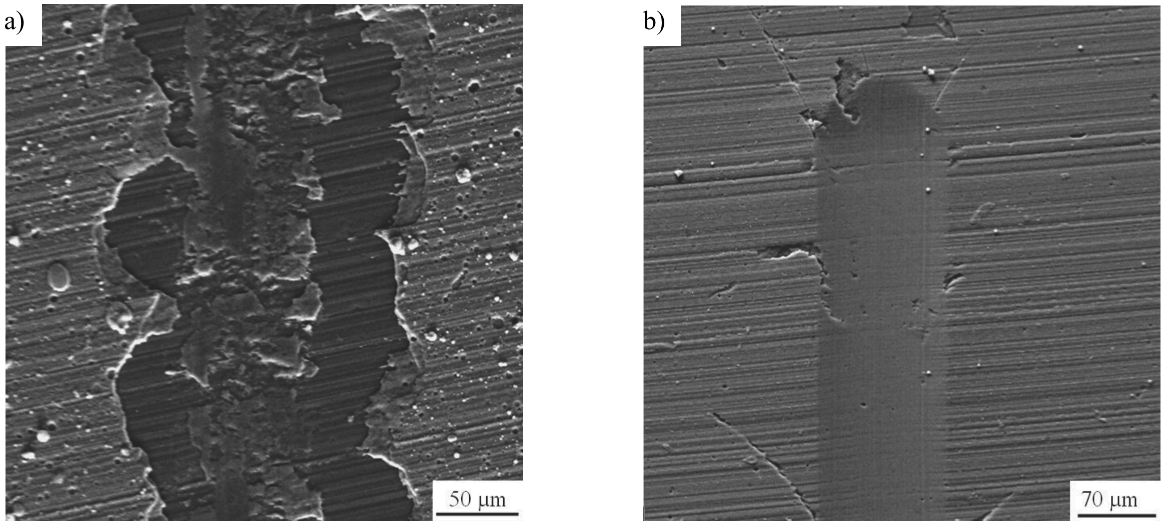

Fig. 10. Characteristic failure of the a) Ti(C,N) and b) (Al,Ti)N coatings deposited on sialon tool ceramics developed during the adhesion scratch test

The identification of coating defects occurred during an investigation of the adhesion using the scratch method was carried out through the observation on the scanning electron microscope. The investigation result is presented in Fig. 10. Three types of dominating damage mechanisms, which are accompanied to a lesser degree by other phenomena are confirmed by the investigation. Firstly, the basic damage mechanism of coatings observed after exceeding the critical load is one-sided and two-sided delamination, which principally involves the coatings obtained on the substrates performed from sintered carbides of the type (Al,Ti)N as well the Ti(C,N) coatings obtained on sialon ceramics. Secondly, a total delamination is a dominant damage mechanism which involves coating of the type Ti(C,N) obtained on the substrate performed from sintered carbides. The abrasion accompanied by cohesive fractures of the coatings and slight chipping and flaking is another damage mechanism found only in the case of coatings obtained on sialon ceramics of the type (Al,Ti)N. It was found that the coating was not ruptured even with maximum load, which is 200 N, but there were only a few cohesive defects and slight chipping in it (Fig. 10b).

In a Table 3 and Fig. 11 is presented the dependence of wear band VB on the tool flank on the machining time T. The investigation has a comparative character. The highest operating durability T=72 minutes of the inserts performed from sialon ceramics was carried out for the cutting edge covered by the (Al,Ti)N coating, however the lowest durability of the cutting edge T=9 minute on the same substrate was exhibited by the Ti(C,N) and (Ti,Al)N coatings. The durability of the cutting edge performed from sialon ceramics without the coatings was estimated at T=11minutes, what means that the (Al,Ti)N and (Al,Cr)N coatings have the influence on the rise of durability of the sialon cutting edge. The highest influence on the durability of the cutting edge T=60 minutes has the (Ti,Al)N coating in the case of inserts performed from sintered carbides. All coatings performed from sintered carbides increase the durability of the cutting edge since the durability of the non-covered tool is T=2 minutes.

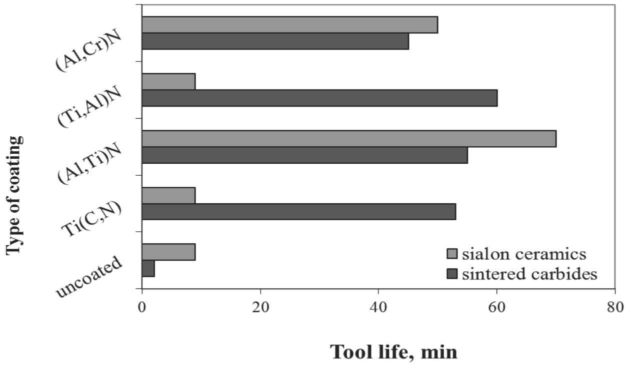

Fig.10. The comparison of tool life T of investigated inserts

Table 3.
Tool life of investigated multi-point inserts

Type of coating	Tool life T, min	
	Sintered carbides	Sialon ceramics
uncoated	2	11
Ti(C,N)	53	9
(Ti,Al)N	60	9
(Al,Ti)N	55	72
(Al,Cr)N	45	50

5. Summary

The improvement of utilitarian properties sintered cutting edges intended to a dry machining with high speed is possible among other things using a surface treatment by applying hard, wear resistant coatings manufactured by PVD and CVD methods. The huge success of both PVD and CVD coatings depends on obtaining thin coatings consist of nitrides and carbides. Also oxides mainly transition metals have an effect on decrease a friction efficient and the improvement of tribological contact conditions in the contact area tool-machined object, increase surface microhardness at edge as well protect against the adherence and diffusion wear. That is why the interest in these types of coatings in the industrial and scientist centers is a very large, but the violent development of these coatings had followed during the last 20 years. However, it is not enough to manufacture a coating material about a hight hardness and low coefficient of friction, but it is a need to optimise the type of coating material in order to among many demands assure of high adherence a coating to surface.

In a case of coatings contain AlN phases about hexagonal lattice, in which occur interstitial bonds, as in a ceramic substrate was obtained a good adherence to substrate performed from sialon ceramic. It means that a type of interatomic bonds occur in the material of both substrate and coating has a great influence on the coating-substrate adhesion. This attention can be extremely helpful when selecting the coating material on ceramic cutting edges since the deposition of coatings on cutting edges in PVD processes is difficult due to their dielectric properties, because without the possibility to polarize the substrate during the deposition process it is difficult to obtain coatings which would have good adhesion to ceramic substrates.

Good adhesion onto booth substrates and PVD coating presents good adhesion onto sintered carbides substrate, this is connected with adhesive-diffuse mechanism of adherence of these coatings to substrate. This is confirmed by presence of transient zone, which is shown in diagrams of changes in chemical elements concentration obtained by GDOES technique.

The exploitative hardness coated cutting edges shows distinct dependence on adherence investigated coatings to substrate. Coatings about good adherence to substrate are characterized by good exploitative properties compared to coatings show weak adherence to substrate are characterized low exploitative properties, in spite of having high hardness.

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DEVELOPMENT FUND

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