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STRUCTURE AND PROPERTIES PVD AND CVD COATINGS DEPOSITED ONTO EDGES OF SINTERED CUTTING TOOLS

STRUKTURA I WŁASNOŚCI POWŁOK PVD I CVD NA OSTRZACH SPIEKANYCH NARZĘDZI SKRAWAJĄCYCH

In papier were presented some results of structure and properties research Ti(C,N) coating by obtained by PVD method and Ti(C,N)+TiN coating obtained by CVD method deposited onto substrate sintered carbides WC-Co type and sialon tool ceramics. Cutting tools deposited investigating coatings are characterized a huge hardness (2442 ÷ 3009 HV0.05) and high machinability in comparison with edges did not cover at all. There were used following searching techniques such as: observation of fractures and a topography of surface into the scanning electron microscope, analysis chemical composition by GDOES method, phase composition was determined by means of the X-ray diffractometer: standard and grazing incidence X-ray diffraction methods in order to characterize some finding structures into a performed work. The mechanical properties were determined on basis of following research: a measurement of hardness by Vickres's method, a measurement of roughness, adhesion by Scratch Test method. Cutting ability were defined on basis of technological cutting trials.
Keywords: Surface engineering, PVD, CVD, Machining, SiAlON

W pracy przedstawiono wyniki badań struktury i własności powłok Ti(C,N) wytworzonej techniką PVD oraz Ti(C,N)+TiN uzyskanej techniką CVD na podłożach z węglików spiekanych typu WC-Co oraz sialonowej ceramiki narzędziowej. Ostrza narzędzi pokryte badanymi powłokami cechują się dużą twardością (2440 ÷ 3000 HV0,05) oraz wysoką skrawnością w porównaniu z ostrzami niepokrytymi. W przedstawionej pracy w celu scharakteryzowania struktury uzyskanych powłok zastosowano następujące techniki badawcze: obserwacje przełomów i topografii powierzchni na skaningowym mikroskopie elektronowym (SEM), profilową analizę składu chemicznego w spektrometrze optycznym wyładowania jarzeniowego (GDOES), jakościową analizę składu fazowego metodą dyfrakcji rentgenowskiej w układzie goniometrycznym oraz w geometrii stałego kąta padania (SKP). Własności mechaniczne określono na podstawie następujących badań: pomiar twardości metodą Vickersa, pomiar chropowatości, przyczepność metodą Scratch Test. Własności użytkowe określono na podstawie technologicznej próby toczenia żeliwa szarego.

1. Introduction

The application of surface treatment technology of tool materials in physical PVD and chemical CVD vapor deposition to obtain modern, both multilayer and gradient coatings about a good wear resistance, also in a high temperature lets into improvement properties these materials in an environment machining among other things: in the consequence of decrease a coefficient friction, improvement conditions of tribological contact in a region of tool contact – worked object, as well protection from both an adhesive and diffusive wear, also an oxidation [1-6].

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 [7, 8]. 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-Si3N4, whereas chemical properties respond to aluminum oxide Al_2O_3 [9, 10].

The research results of PVD and CVD coatings were

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presented in this paper, which having in purpose to decrease an operating hardness of machining edges from sintered carbides and a modern sialon tool ceramic.

2. Materials

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

3. Research methodology

The thickness measurement of investigated coatings was carried out by "Kalotest" method (Table 1).

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 0,49 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.

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 L_c = 0,8 mm with an accuracy of ±0,02 μm. The R_a parameter was accepted as quantity describe a roughness.

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 100 N (for a PVD coatings) and from 0 to 200 N (for a CVD coatings) 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.

Observation of coatings structure and surface topography was carried out by use of 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.

Phase composition analyses of investigated samples were made on the PANalytical X'Pert PRO diffractometer, working in goniometer system (using the filtered X-ray Co $K\alpha$, step 0.05, time of counting 10 sec.) at the voltage of 40 kV and tube current of 30mA. Moreover, phase analysis by Grazing Incidence X-Ray Diffraction (GIXRD) method was performed.

Machining properties of investigated samples were determined by technological cutting trials. The tests of cutting ability were carried out in dry machining condition, without cutting fluid. The grey cast iron was used for the continuous turning. During the cutting process, the mean width of flank wear VB_B was measured. The tests were stopped when the VBB value obtained the assumption criterion VB_B = 0.20 mm. Tool life T is determined by time of continuous machining (determined by minutes) close to a threshold VB_B. Operations were carried out with a cutting speed v_c of 180 m/min; feed rate f_z=0.2 mm/rev tooth; depth of a cut a_p=1 mm.

4. Results and discussion

In the consequence of measurements of thickness coatings it was found that the same type of coatings onto different substrates are shown another thicknesses (Table 1.). In both causes a coating deposited onto a sintered carbide substrate is thicker rather than on sialon tool ceramic, moreover it was found that in a case of CVD coating – Ti(C,N)+TiN type the difference in thickness is close to 100%.

The thickness difference of CVD coatings onto both substrates is connected with a carbon source in increasing coatings. The CVD coating deposited onto sintered carbides substrates absorbs a carbon from a substrate and as well from a CH_4 gas. However, a carbon source in a rising coating on a sialon tool ceramic is only from a reactive gas CH_4.

TABLE 1

Investigation results of thickness, hardness, roughness and adhesion testing coatings

Substrate	Coatings	Thickness of coating, μm	Hardness	Roughness factor Ra, μm	Critical loading L_c, N
Sintered	–	–	1668 HV0.5	0.06	–
carbides	Ti(C,N)	2,1	3009 HV0.05	0.50	81
WC-Co	Ti(C,N)+TiN	5	2442 HV0.05	0.40	110
Sialon	–	–	1838 HV0.5	0.06	–
tool	Ti(C,N)	1,8	2843 HV0.05	0.38	26
ceramic	Ti(C,N)+TiN	2,8	2746 HV0.05	0.20	78

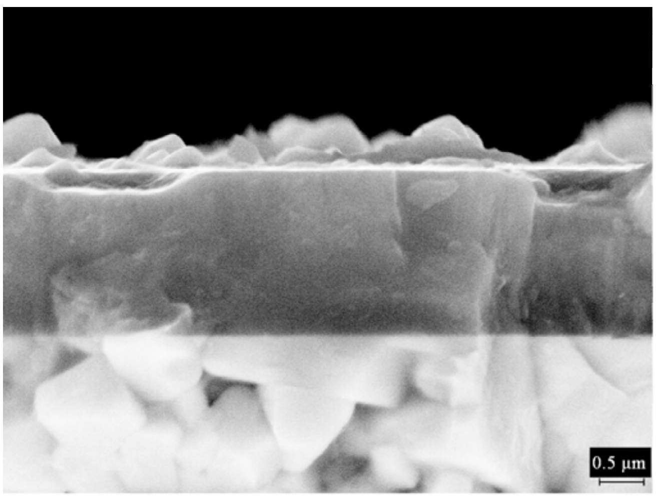

Fig. 1. Fracture of the Ti(C,N) coating deposited onto sintered carbides substrate

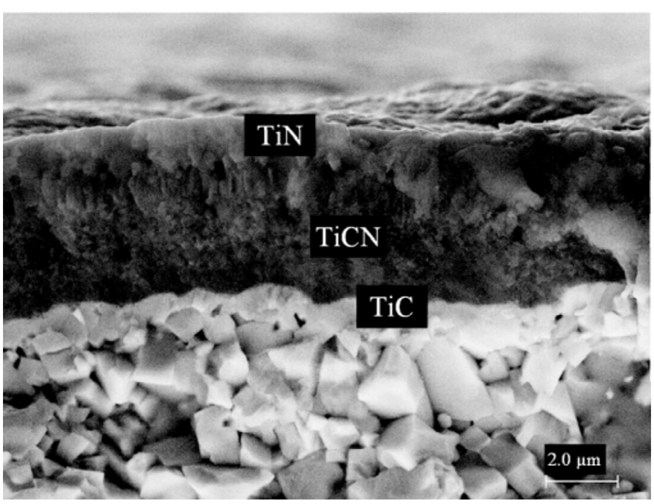

Fig. 2. Fracture of the Ti(C,N) coating deposited onto sintered carbides substrate

As the result of fracture coating observation onto scanning electron microscope it was found that all investigated coatings are characterize compact fibrous structure without pores and well adhere to a substrate, furthermore particular layers coating of Ti(C,N)+TiN adhere tight to each other. It was found that Ti(C,N) coating 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). It was found that a multilayer coating of Ti(C,N)+TiN type, which was obtained by CVD method in a interfacial zone coating - substrate has a thin layer fine fracture phase TiC (Fig. 2), what was proved by XRD method described in a further part. However, a Ti(C,N) layer in CVD coatings is characterized by changing structure in a gradient way from a fine-grained near a substrate is charging progressively in a columnar structure. Observations of surface topography in a scanning electron microscope were found that a surface coatings morphology produced with using a PVD technique onto sintered carbides from WC-Co type and 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. 3). Occurrence of these morphologic defects is connected with a cathode arc evaporation process. However, the Ti(C,N)+TiN surface deposited onto both substrates is clearly differ. This coating deposited on sintered carbide is shown a surface, which is lightly wavy about mild shapes, however a coating Ti(C,N)+TiN on a sialon is composed of grains about spherical shapes and a size about 2 μm. All these inhomogeneity observed in SEM cause an increase of roughness surface in comparison with uncoated inserts.

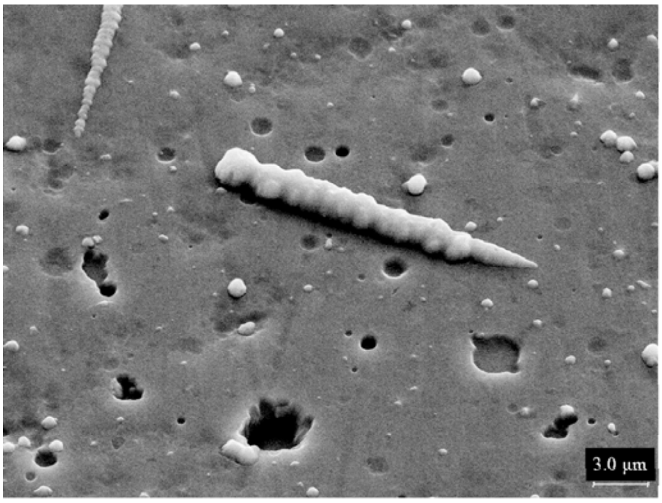

Fig. 3. Surface topography of Ti(C,N) coating deposited on sialon tool ceramics substrate

The chemical composition analysis by GDOES method is shown that changes of atomic concentration of coating elements and a material substrate (Fig. 4). The modification character of concentration elements forming coatings is shown their gradient build.

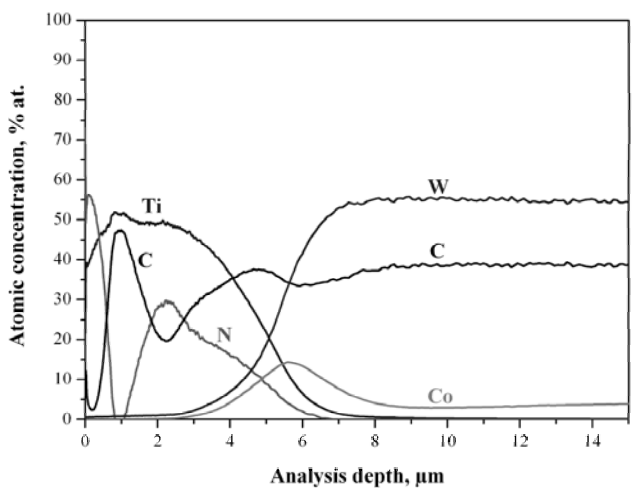

Fig. 4. Changes of constituent concentration of the Ti(C,N) and the sintered carbides substrate materials

The phase composition analysis with using a X-ray diffraction method is shown that in investigated coatings are presented some expect phases. PVD coating contains a titanium carbonitride (TiCN) about a fcc lattice (fig. 5). However, in a case of CVD coating – Ti(C,N)+TiN type, the diffraction analysis in a goniometric system is shown occurring both TiC, TiN and TiCN phases. Furthermore, in all cases except a Ti(C,N)+TiN coating deposited onto sintered carbides substrate was observed some peaks come from a substrate. This occurs from a small thickness of coatings, smaller than a depth of penetrating X-ray. Additionally, performed a research by SKP method for ? equaled 0,5 °, 3 ° and 15 ° (fig. 6) multilayer coatings PVD is confirmed a complex order occurring phases.

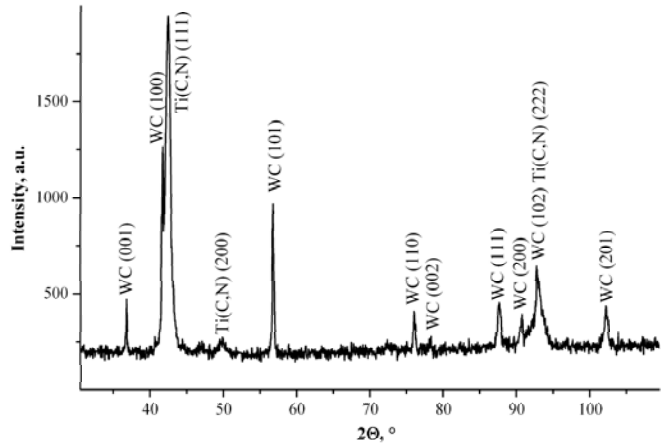

Fig. 5. X-ray diffraction pattern of Ti(C,N) coating deposited on sintered carbides

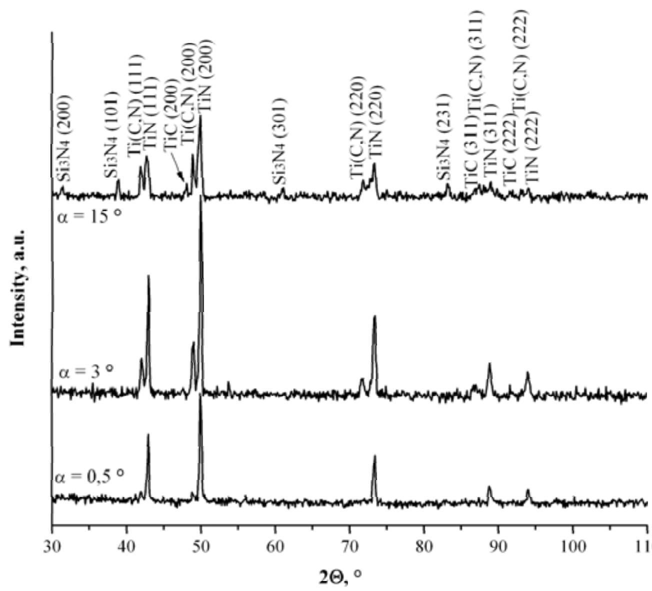

Fig. 6. X-ray diffraction pattern of Ti(C,N) coating deposited on sialon tool ceramics obtained by GIXRD method

The analysis of glancing incidence X-ray diffraction method for α equaled 0,5 ° is presented existing on a surface only TiN phase, increasing a glancing incidence X-ray, it was observed occurring additionally peaks from a Ti(C,N) phase, however with the highest angles were presented additionally peaks from titanium carbide, what proves about exiting in these coatings a transient phase TiC observed on fractures in a scanning electron microscope.

In the consequence of hardness measurements investigated uncoated samples and coated with PVD and CVD coatings it was found a increase surface hardness after deposited coatings (Table 1). It was also found that a Ti(C,N) coating is shown higher hardness than Ti(C,N)+TiN coatings. There was observed also that coatings deposited onto sintered carbides are harder than coatings from the same type deposited onto sialon tool ceramic.

The results of the scratch test measurement for the studied coatings is revealed that coatings present better adhesion to a sintered carbides substrate than sialon tool ceramic substrate (Table 1). In the case of PVD coating this should be connected with a character of deposited process and impossibility polarization of ceramic substrate. The higher adhesion to sintered carbides and a difference in thickness in a coating obtained from CVD method is caused by diffusion a carbon from sintered carbon substrate during depositing process, what causes a form connection about a adhesive – diffusivity character, but not adhesive as in a case of ceramic substrate. Furthermore, it was fund that a better adhesion is presented a Ti(C,N)+TiN coating than Ti(C,N), what also should be connected with a character of interfacial phase of coating-substrate.

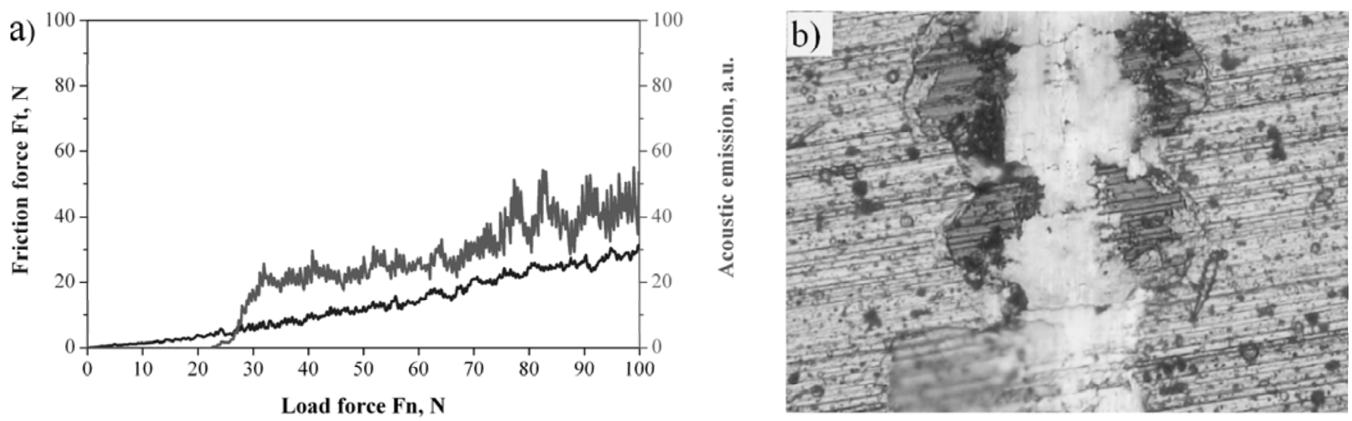

Fig. 7. (a) Acoustic emission (AE) and friction force Ft as a function of the load force Fn for Ti(C,N) gradient coating on sialon tool ceramics; (b) scratch failure at L_c (opt) = 26 N, mag. 200x

TABLE 2
Results of technological cutting trials uncoated and coating inserts

Coating	Tool life T, min	
Sintered carbides Sialon tool ceramic–	2	11
Ti(C,N)	52	9
Ti(C,N)+TiN	27	15

As a result of performed technological cutting trials of gray cast iron investigated coated and uncoated inserts, it was found that a Ti(C,N)+TiN coating deposited also on sintered carbides and sialon tool ceramics improves a cutting ability. However, a Ti(C,N) coating improves considerably a cutting ability of tool from sintered carbides, but it does not increase a cutting ability of sialon tool ceramic (Table 2, Figs. 8, 9). A insert from sintered carbides, deposited by PVD coating of Ti(C,N) type for which the life time is equal 52 minutes, shows the highest cutting ability. The observation of edges were shown that the most often cause of wear was a surface abrasion of both flank and top rake face, after machining on scanning electron microscope. In a case of edge from sialon tool ceramic coated Ti(C,N), it was found that a coating was spalled onto a tool flank surface after 9 minutes of machining (fig. 10), what a correlates with a low adhesion this coating to surface. Additionally, the metallographic observations show that creating a built-up edge from a machining material on tools from sintered carbides, what confirms that the chemical composition analysis by EDS method from micro areas.

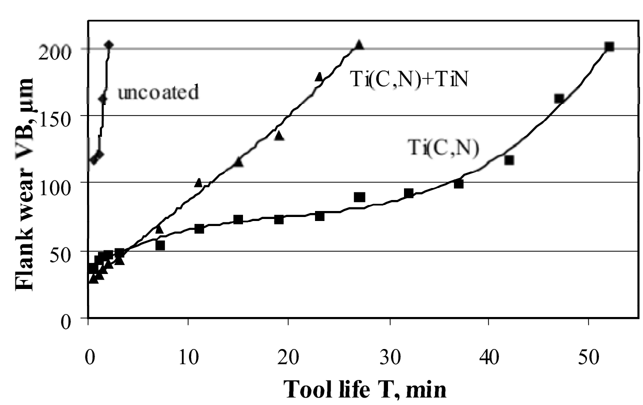

Fig. 8. Wear plots for uncoated and coated inserts from sintered carbides

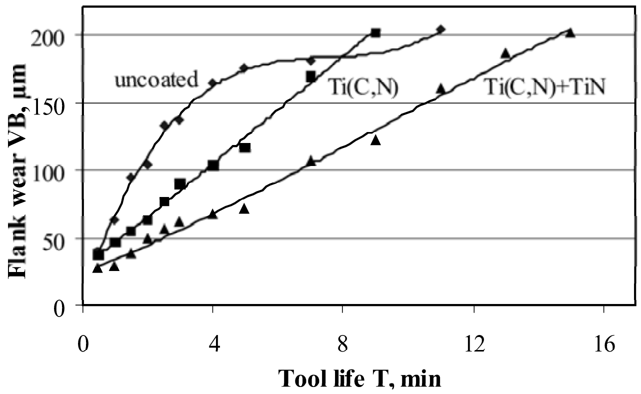

Fig. 9. Wear plots for uncoated and coated inserts from sialon tool ceramics

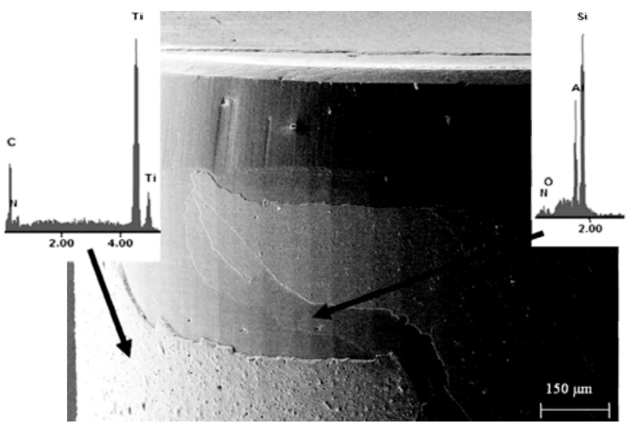

Fig. 10. Characteristic wear of tool flank of sialon inserts coated by Ti(C,N) coating, EDS spectrums from microareas

As a result of work surface roughness of gray cast iron, after machining with using testing inserts, it was found that protective coatings influence on quality improvement of working surface (Table 3). Moreover, it was found that better quality of work surface was getting as a result of machining deposited inserts from a sialon ceramic rather than from sintered carbides.

TABLE 3

Quality of work surface gray cast iron after machining

Coating	Roughness of work surface R_a, µm	
	Sintered carbides	Sialon tool ceramic
–	4.55	3.03
Ti(C,N)	4.16	2.34
Ti(C,N)+TiN	4.41	2.09

5. Summary

On the basis of carried out investigations, it was found that:

- The high hardness and a good adhesion of coating: Ti(C,N)+TiN obtained on both substrates and a Ti(C,N) deposited onto sintered carbides influence on period increase of cutting ability tools, as well they enable apply a dry machining without a liquid lubricating – cooling, what is a very important pro-ecological aspect.
- The mechanical properties of coating, mainly a hardness depend on a getting structure. The Ti(C,N) coating show a fine-crystalline structure, which was classified to a IV (T) zone of the Thornton model, what influenced on bigger hardness of this coating in comparison to a Ti(C,N)+TiN coating about columnar crystallites.
- The structure of obtaining coating strongly influences on mechanical properties deposited tools. It was found that a Ti(C,N) coating about a fine-grained structure is presented a better cutting ability, although a worse adhesion to substrate from a Ti(C,N)+TiN coating on the same substrate pointing out a better adhesion, but a bigger crystallite.
- Both the kind of used substrate and a deposition method influence on adhesion coating to substrate. The investigations were shown that a CVD coating has a good adhesion both to a substrate from sintered carbides and as well to sialon tool ceramic, what influence on improvement of good adhesion to sintered carbides, whereas a weak to applied sialon tool ceramic.

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