Characteristics of Ti(C,N) and (Ti,Zr)N gradient PVD coatings deposited onto sintered tool materials

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Manufacturing and processing

ABSTRACT

Purpose: The purpose of this paper is the characterisation of Ti(C,N) and (Ti,Zr)N thin films produced by cathode arc evaporation physical vapour deposition (CAE-PVD) method onto sintered carbides and sialon tool ceramics.

Design/methodology/approach: The GDOS spectrometer was used to indicated chemical concentration changes of the coating components. The phase composition of the investigated coatings was determined by means of the X-ray diffractometer: standard and grazing incidence X-ray diffraction methods. Observations of surface topography and fractures of the deposited coatings were carried out in the scanning electron microscope. Tests of the coatings adhesion to the substrate material were made by use of the scratch test. The microhardness of substrates and coatings were measured by Vickers method. Cutting ability was determined by technological cutting trials.

Findings: The studied PVD gradient coatings deposited by cathodic arc evaporation method are demonstrated high hardness, adhesion and wear resistance. The critical load LC2, which is in the range 21-53 N, depends on both the coating type and material substrate. Coatings deposited on sintered carbides are extending tools life.

Practical implications: Ti(C,N) and (Ti,Zr)N gradient coatings can be applied for cutting tools.

Originality/value: It should be emphasized, that the mechanical properties of the PVD coatings described in this work are very encouraging and therefore their application for products manufacturing at mass scale is possible in all cases where reliable, very hard and abrasion resistant coatings, deposited onto sintered tools substrate are required.

Keywords: Thin&thick coatings; Mechanical properties

1. Introduction

Machining is commonly applied and this makes a necessity of search new solutions for tool materials in order to raise a tool efficiently and its use properties. Modern sintered tool materials, among of them: sintered carbides and tool ceramics, are determined a group of tool materials, which has delighted a huge interest in industrial and scientific environments, concerned machining problems. Development of PVD method was caused a

large progress in field of tool materials and their wear resistance, mainly tribology. These coatings are delighted a decline interest and their development and researches proceed in two layers. On the one hand, coatings development from monolayer, through multilayer up to gradient coatings. Second direction of researches for wear resistance coatings concerns an expansion of these coatings for bigger and bigger number of different kind substrates [1-9]. Coating deposition a substrate conducted electric current doesn't offer considerable difficulties, but covering no conducted materials is questionable. Investigations carried out by G.E. D'Errico et al. [10,11] in application of PVD coatings onto insert ceramics from cermets indicated that these coatings don't improve on wear resistance of these tools besides in some cases even they are worsen much inability multi-point inserts. On the other hand, researches conducted in the future time by many researchers [1,3-5,12,13] mainly onto Si_3N_4 and Al_2O_3 base from ceramic, as well cermets and ceramic indicated with whiskers are shown a positive influence of PVD on tool life.

2. Materials

The examinations were carried out on the multi-point inserts uncoated and coated by Ti(C,N) and (Ti,Zr)N gradient PVD coatings. SNGN 120412 type inserts, which were made by both types of materials: sintered carbides WC-Co and SiAlON tool ceramics were used. The substrates were ultrasonic cleaned in pure acetone and exsiccated in dry heat stream, before they were put inside of a working chamber of PVD device. First substrates were electron heated, next they were ion cleaned in vacuum chamber. The coatings deposition process was made with use of a device based on the cathodic arc evaporation (CAE) method. Cathodes, which are containing pure Titanium metal and TiZr (80:20 at.%) alloy were used for coatings deposition. The basic pressure was 5×10^{-6} mbar, the temperature deposition was approximately 350 °C. In case of Ti(C,N) gradient coating, a change of nitrogen and carbon concentration by both controlled active flow gases: N_2 and C_2H_2 were obtained (Fig. 1). The current of three Titanium targets was constant and equal to 60A. Three targets: one pure Titanium and two TiZr alloy to produce (Ti,Zr)N gradient coating were used. During deposition process of (Ti,Zr)N gradient coating, both of Zirconium and Titanium concentration were modified, which was caused by controlled evaporation current intensity of Ti of and TiZr targets (Fig. 2). The intensity of Nitrogen gas flow was 250 sccm. The flow of non-reactive gas – Argon – was equal to 40 sccm for Ti(C,N) coating process and 10 sccm for (Ti,Zr)N coating process. During the deposition process bias voltage was equal to -100V. Due to non-conductive SiAlON substrate this inserts were put down on a steel target, which was connected with bias. Sample holders were set in the rotatory movement.

3. Methodology

The chemical concentration of the coating components has changed, both perpendicular to their surface and in the transition zone between the coating and the substrate material were evaluated in tests carried out in the GDOS-750 QDP glow

discharge optical spectrometer from Leco Instruments. The following operation conditions of the spectrometer Grimm lamp were fixed during the tests: lamp inner diameter– 4 mm; lamp supply voltage – 700 V; lamp current – 30 mA; working pressure – 100 Pa.

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 Kα, 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 method was performed.
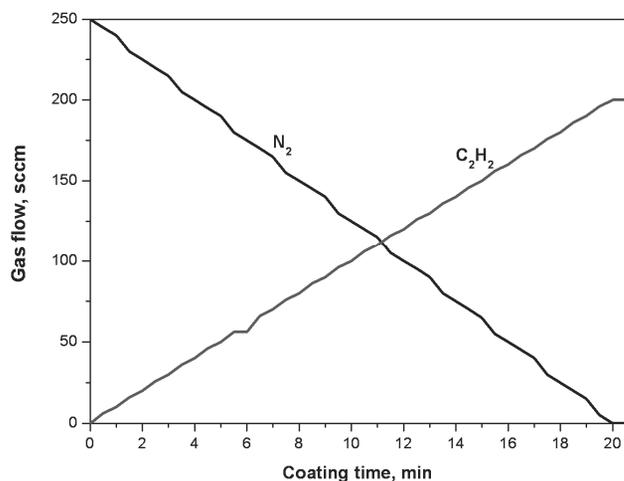
Fig. 1. Change concentration of reactive gases flow during Ti(C,N) coating deposited process
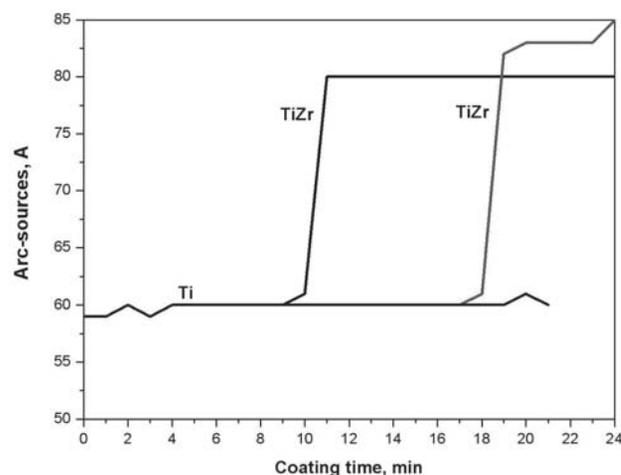
Fig. 2. Current intensity sputtering of Ti of and TiZr targets during (Ti,Zr)N coating deposited process

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 15-20 kV.

To obtain hardness coatings the Dynamic Ultra Microhardness Tester DUH 202 from Shimadzu Corporation was used. The Vickers indenter has a load of 50 mN, which provides, that the penetration depth will not exceed 10% of coatings thickness. This eliminates the influence of substrate hardness on measurement result.

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, load increase rate (dL/dt) 100 N/min, indenter's speed (dx/dt) 10 mm/min. The critical load L_C was determine by acoustic emission level (AE) and observation of failure at the scratch.

Operating 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 put for the continuous turning. During the cutting process, the mean width of flank wear VB_B was measured. The tests were stopped when the VB_B 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

The chemical composition analysis was made by glow discharge optical emission spectroscope method showed that all coatings include titanium and nitrogen, moreover, the Ti(C,N) coating had contained considerable quantity of carbon and (Ti,Zr)N coating contain Zirconium. Figure 3 shows the change of atomic elements concentration in function of distance from TiCN coating surface deposited onto sintered carbides WC-Co. The change of nitrogen and carbon concentration in Ti(C,N) coating corresponding with the concentration change of reactive gases flow during deposition process. Gas flow N_2 is initially on high level (Fig. 1) and it is decreasing during deposition process whereas C_2H_2 reactive gas is increasing from 0 sccm initially to 200 sccm at the end of the process. The GDOES analysis revealed evident gradient change concentrations of N and C elements. According to assumption, the nitrogen concentration is high from substrate and decreasing to zero together with the coating growth and carbon concentration is increasing on cross-section of coating from substrate. In case of (Ti,Zr)N coating gradient change concentration of elements were determined by the change of

sputtering current intensity of Ti of and TiZr targets during deposition process for this coating (Fig. 2). The change concentration of titanium and zirconium is obvious in Figure 4. Gradient changes of nitrogen concentration was observed too.
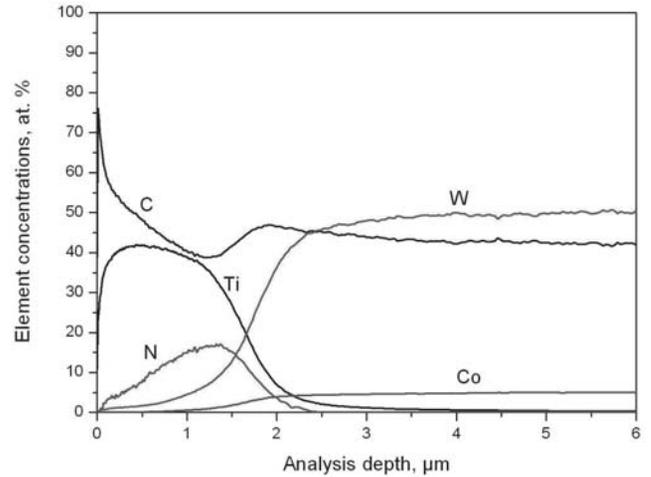
Fig. 3. Constituent concentration changes of the TiCN and the substrate materials
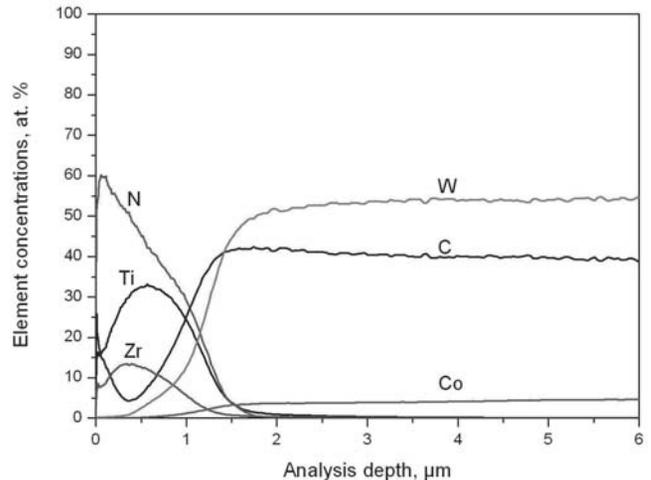
Fig. 4. Constituent concentration changes of the TiZrN and the substrate materials

Table 1.
Specification of substrates and coatings investigated in the work

Substrate	Coating	Hardness, GPa	Critical load L_C, N
Sintered carbide	Uncoated	16.4	-
	Ti(C,N)	31.2	53
	(Ti,Zr)N	29.6	52
SiAlON ceramics	Uncoated	18.0	-
	Ti(C,N)	29.7	35
	(Ti,Zr)N	28.6	21

In order to obtain more precise information from surface layer analyzed materials further researches was applied grazing incidence X-ray diffraction method with use of the parallel beam collimator, which is located before proportional detector. These researches were carried out due to the overlap peaks of the substrate as well as from coating, and the peaks intensity (Fig. 5), what makes the analysis of received results difficult. Due to the possibility of XRD pattern registration, by grazing incidence X-ray diffraction on the sample surface, it exists the possibility of receiving the XRD patterns from thin layers in result of volume enlargement of examined material, which is participate in diffraction. On analysed XRD patterns obtained by grazing incidence X-ray diffraction only reflexes coming exclusively from studied coatings were identified. Figure 6 presents X-ray pattern for this method from the same sample as Figure 5. Investigations have indicated the existence of only one f.c.c. phases in all examined cases. It was confirmed, that according to the
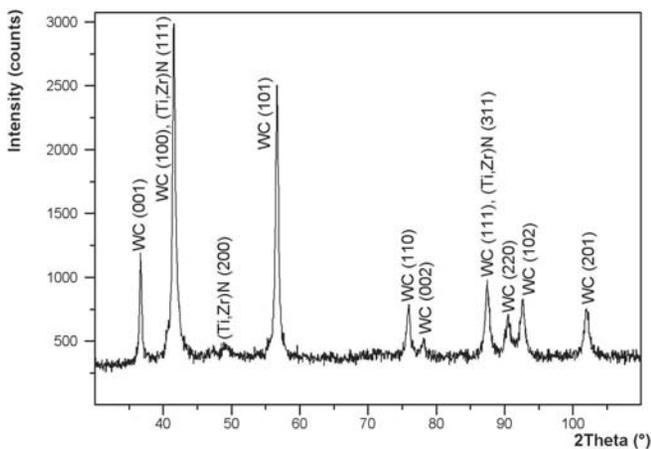
assumptions, coatings are containing TiCN phase for Ti(C,N) coating and TiZrN phase for (Ti,Zr)N coating.

Fractography based on investigation results confirmed, that produced coatings are evenly deposited on substrates the average thickness of 2 μm. Fracture observations of the coatings by SEM shows, that deposited coatings structures are corresponded to IV zone (T) of the Thornton model (Fig. 7a). Ti(C,N) coating are characterized by fine granular fracture. (Ti,Zr)N coating is characterized by compact fibrous structure. Both coatings are shown fracture without discontinuity and tight adherence to the substrate. This structures are characterized by high hardness, higher then uncoated substrates (Table 1). Unevennesses of both coatings surfaces on both substrates were observed. This is due to micro-drops from metal sources evaporation during deposition process (Fig. 7b). Chemical analysis of this drops by energy dispersive spectroscopy method proved the existence of titanium and also zirconium in case of drops on (Ti,Zr)N coatings.

Fig. 5. X-ray diffraction pattern of TiZrN coating deposited on sintered carbide
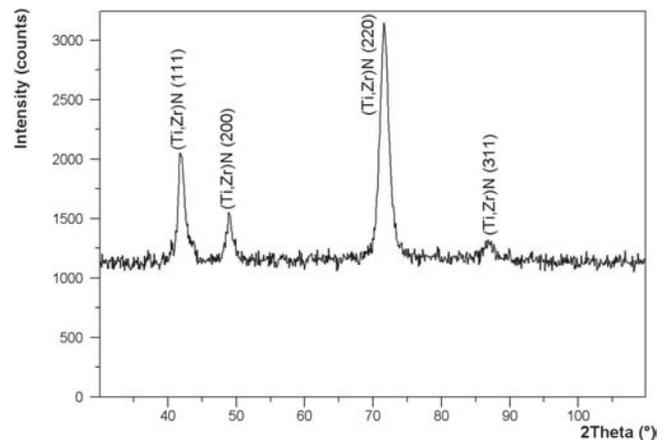
Fig. 6. Grazing incidence X-ray diffraction pattern of TiZrN coating deposited on sintered carbide (α=2)
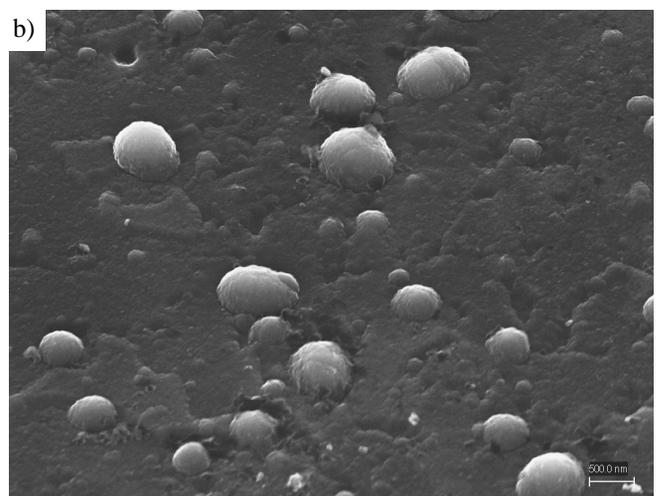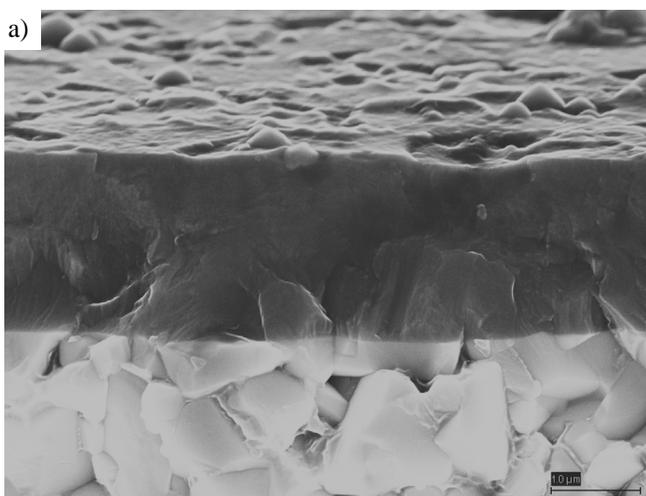
Fig. 7. SEM images from Ti(C,N) coating deposited onto cemented carbide, a) fracture, b) topography surface

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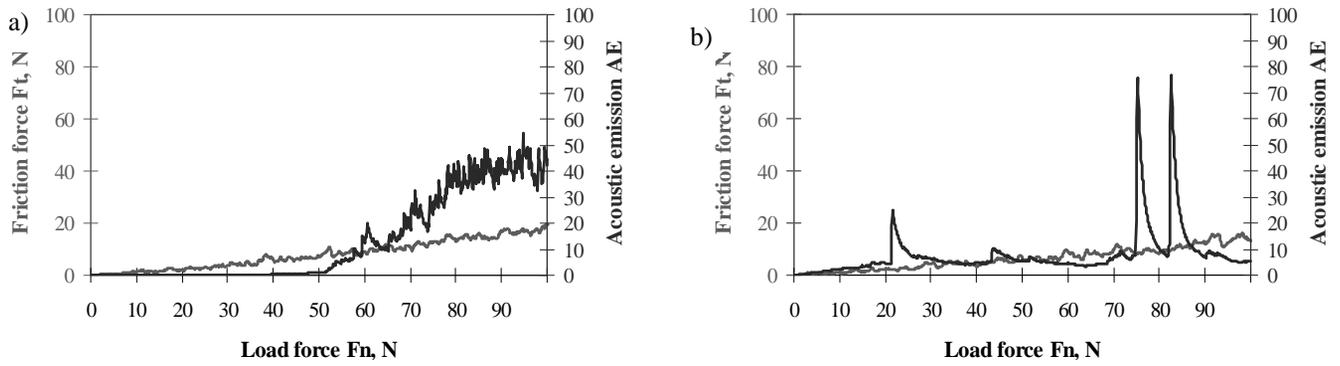
Fig. 8. Acoustic emission (AE) and friction force Ft as a function of the load Fn for (Ti,Zr)N coating on: a) sintered carbide; b) sialon
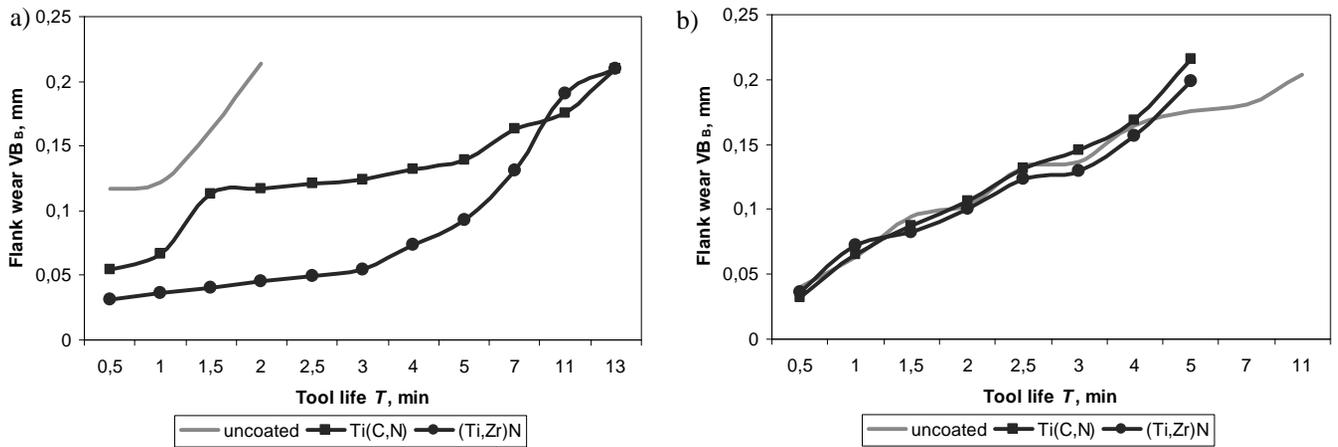
Fig. 9. Wear plots, substrate: a)sintered carbides; b) sialon tool ceramics
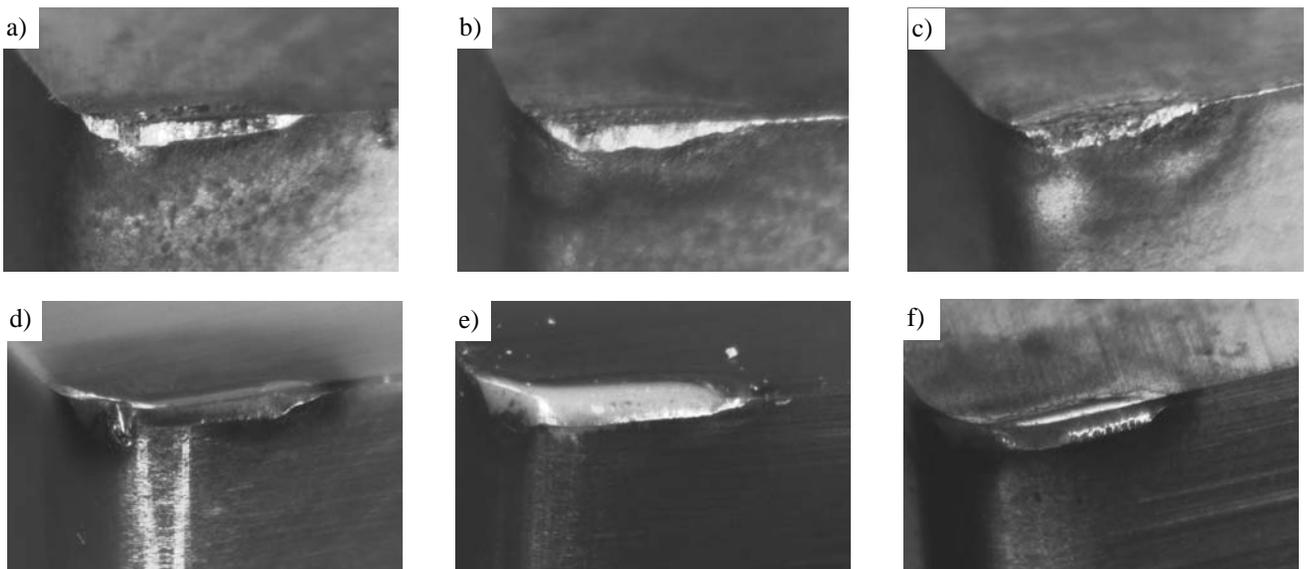
Fig. 10. Characteristic wear of tool flank of inserts: a) uncoated sintered carbide, b) TiCN coating on sintered carbide, c) TiZrN coating on sintered carbide, d) uncoated sialon ceramic, e) TiCN coating on sialon ceramic, f) TiZrN coating on sialon ceramic

Adhesion of examined coatings to substrates is characterized by the critical load L_{C1} (Table 1). This value was determined with use of the scratch test with the linear growth load. The friction force Ft and acoustic emission AE as a function of the load Fn during scratch testing were registered (Fig. 8). The critical load L_C is a point on the curve of the friction force, where the damage of the coating was observed and corresponding to acoustics emission signal was registered. Scratch adhesion tracks were analyzed using the light microscope coupled with a measuring gauge. Thus the values of the critical load L_C could be obtained on the metallographic observations basis. Critical load L_C all coatings to sintered carbides surfaces indicated on good adherence. L_C values booth coatings are above 50 N. But adhesion of this same coatings deposited onto SiAlON tool ceramics are considerable lower. Values in this case are equal between 21 and 35 N. Probably this is caused by too low kinetic energy of particles, which were created the coating during deposition process, and this in the turn is caused by the bias lack for nonconductive ceramics substrate. To tell the truth, in order to improve adherence for these coatings, sialon substrates were put down on a steel target, which was connected with bias, but this solution appears to be insufficient.

In order to determine wear resistance properties of deposited coatings, technological cutting trials was made. The investigation has the comparative character. This means, that the criterion, which determines the tool life was the time T of cutting grey cast iron measured until the width of flank wear VB_B=0.20 mm was achieved. Figure 9 is presented a comparison between the T times for inserts uncoated and coated by examinated coatings. The coatings deposited onto cemented carbides increasing tool life, which corresponded with high hardness and good adhesion of this coatings to substrate. In case of coated sialon tool ceramics the low cutting ability is caused a possibility due to weak adherence. Figure 10 is showed wears of tool flank after machining.

5. Summary

Changes of the investigated coatings chemical composition corresponds with deposition parameters and their determined by those. XRD diffraction patterns confirmed, as presumed, than deposited coatings Ti(C,N) and (Ti,Zr)N contained one fcc phase only. The fracture observations by SEM indicated, that columnar structure exists, which may be considered as compatible with the Thornton model (zone IV). Upon examination of the thin films, it was found, that the coatings were composed of fine crystallites without discontinuity. Adhesion test indicated better connection of coatings with sintered carbides than with sialon tool ceramics. Critical load L_C for coatings deposited on sintered carbides is above 50 N but L_C for sialon substrate in between 21 and 35 N. Deposited and investigated coatings are improved cutting ability for sintered carbides inserts but there is not upgrade of wear resistant for sialon tool ceramics. However the hardness value of all coatings is high, between 28 and 31 GPa.

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