HARD, WEAR RESISTANT COATINGS DEPOSITED ON SINTERED TOOL MATERIALS

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Abstract. *These days, the security margins are so tight, that even our safety depends on the quality of the materials that we use every day, so it is very important to be able to manufacture parts at high level of productivity and quality. Parallel to the materials development there was also the improvement of tool materials, especially ceramic tool materials. The most popular kinds of ceramic tool materials are: the oxide Al_2O_3 ceramic and nitride Si_3N_4 based ceramic. The paper presents investigation results of structure and properties of the coatings deposited with the PVD and CVD techniques on cutting inserts made from sintered tool materials. The investigation includes the metallographic analysis on the transmission and scanning electron microscope, chemical composition analysis as well as the analysis of the mechanical and functional properties of the material. Tests were carried out on the multipoint inserts made from the ceramic tool materials based on Al_2O_3 and Si_3N_4, uncoated and coated with gradient, multicomponent and multilayer hard wear resistant coatings with PVD and CVD processes. It was demonstrated, that adhered closely to each other were characterized by good adhesion to the substrate. It was demonstrated, basing on the technological cutting tests of grey cast iron, that putting down onto the tool ceramics the thin anti-wear coatings in the PVD and CVD processes increases their abrasion wear resistance, which has a direct effect on extending the tool edge life. Basing on the roughness parameter R_a of the machined cast iron surface after the cutting tests, improvement was revealed of the machined material properties, cut with coated oxide ceramics compared to material machined with the uncoated tools.*

Keywords*: Sintered tool materials, PVD, CVD, SEM, TEM.*

1. Introduction

An interest is growing in the last years in particular in the ceramic and ceramic-carbide cutting materials used mostly for machining of cast iron and steel at high cutting speeds. The Al_2O_3 based oxide and Si_3N_4 based nitride tool materials feature the biggest and dynamically growing group of materials among the ceramic tool materials [6, 9].

These days, the security margins are so tight, that even our safety depends on the quality of the materials that we use every day, so it is very important to be able to manufacture parts at high level of productivity and quality. Parallel to the materials development there was also the improvement of tool materials, especially ceramic tool materials. The most popular kinds of ceramic tool materials are: the pure oxide Al_2O_3+ZrO_2 ceramic and the Si_3N_4 (silicon nitride) based ceramic [1, 2, 6, 8, 10].

The pure oxide Al_2O_3+ZrO_2 were thin particles of Al_2O_3 (between 1 and 10 microns) together with ZrO_2 (with the purpose of offering higher tenacity to the cutting tool), is obtained by a cold pressing procedure, but this makes it very porous, so to eliminate those, the material is sintered at a temperature of 1700ºC or more. The major advantages of oxide ceramic are: high hardness at high temperatures, no chemical reaction with steel, high compression resistance and possibility of high cutting speed nevertheless it has also disadvantages like: high brittleness and low thermal shock resistance [1, 6, 10]. The Si_3N_4 (silicon nitride) based ceramic, is a relatively new material (developed around 1970), single-phase Si_3N_4, is a highly covalent compound which exists in two hexagonal polymorphic crystalline forms, α and the more stable β. In comparison to other kind of ceramic tool materials, it presents several improvements, like: better chock resistance, considerable hardness at high temperatures, although it does not present equal chemical stability as the aluminium based ceramics when working with steel, it is excellent to use against grey cast iron, with high removal percentage of material [9, 10].

Employment of the surface treatment technology for tools made from tool materials, with the PVD and CVD methods, to obtain the high wear resistant coatings makes it possible to improve the properties of these materials in the dry-cutting conditions, by – among others – decreasing their friction coefficient, micro-hardness increase, improvement of the tribological contact conditions in the cutting tool-machined workpiece zone, and also to improve protection against the adhesion and diffusion wear [1-10].

The application of thin coatings deposited at PVD and CVD processes on both of the substrates is the possibility to improve the tribological behaviour of the pure oxide Al_2O_3+ZrO_2 ceramic as well as the nitride Si_3N_4 based ceramic.

2. Experimental procedure

Tests were carried out on the multipoint inserts made from the Si_3N_4 nitride ceramics and oxide ceramics, uncoated and coated with hard wear resistant coatings in the PVD and CVD processes (Table 1).

Table 1
Characteristics of the investigated cutting tools

Substrate	Coatings		Coatings thickness, μm	Process type
	Type	Composition		
Si_3N_4	gradient layer	TiN+TiAlSiN+AlSiTiN	3.5	PVD
	double layer	TiN+Al_2O_3	6.0	CVD
Al_2O_3+ZrO_2	gradient layer	TiN+TiAlSiN+AlSiTiN	2.5	PVD
	double layer	TiN+Al_2O_3	10.0	CVD

Observations of surfaces and structures of deposited coatings were carried out on the transverse fractures on the Opton DSM 940 scanning electron microscope (SEM) and on the on the JEOL JSM – 5610 one equipped with X-ray energy dispersive spectrometer (EDS) with the accelerating voltage in the range of 15-20 kV. The diffraction examinations and examinations of thin foils were made on the JEOL 3010CX transmission electron microscope (TEM) at the accelerating voltage of 300 kV. The diffraction patterns from the transmission electron microscope were solved using the computer program.

The X-ray qualitative and quantitative microanalysis and surface distribution analysis of elements in the investigated coatings were made using the EDS X-ray energy dispersive radiation spectrometer, featuring the standard equipment of the scanning microscope.

The microhardness tests using the Vickers method were made on the Shimadzu DUH 202 tester. The load of 70 mN was employed, making it possible to eliminate to the greatest extent the influence of the substrate material on the measurement results.

Adhesion evaluation of the coatings on the investigated inserts was made using the scratch test on the CSEM REVETEST device, by moving the diamond penetrator along the examined specimen's surface with the gradually increasing load. The tests were made with the following parameters: load range 0-100 N, load increase rate (dL/dt) 100 N/min, penetrator's travel speed (dx/dt) 10 mm/min, acoustic emission detector's sensitivity AE 1. The critical load L_C, at which coatings' adhesion is lost, was determined basing on the registered values of the acoustic emission AE and friction coefficient F_t. Additionally L_c was determined optically.

Machining properties of tested materials were defined on the basis of technological tests of the continuous machining of grey cast iron EN-GJL-250 with the hardness of 215 HB. The width of the wear band on the surface of the tool used VB=0.3 mm for precise machining was the main criterion of the cutting edge consumption evaluation. These were the parameters used in the research:

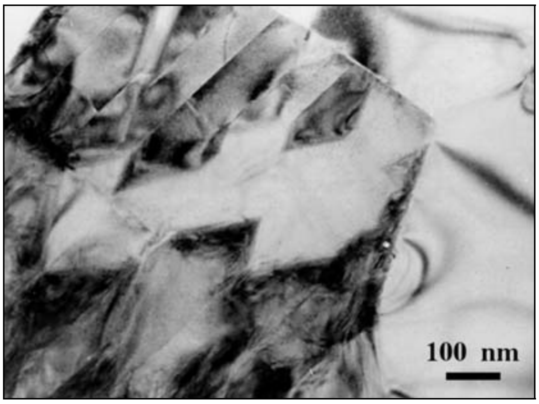
- The rate of feed, f=0.2 mm/turn
- The width of turning, a_p=2 mm
- Machining speed, v_c= 400 m/min

The examination of deposited coatings wear was made on the light microscope and scanning electron microscope (SEM) using several types of magnification. The roughness measurement of the machined surface of the grey cast iron EN-GJL-250 (after the machining process) was made on the TAYLOR-HOBSON's SURTRONIC 10 appliance.

3. Discussion of investigation results

Basing on the examinations of thin foils parallel to the coating surface in the transmission electron microscope it was found out that the Al_2O_3+ZrO_2 oxide tool ceramics contains the aluminium oxide grains with the hexagonal lattice ($P6_3mc$ space group) and the ZrO_2 ones with the monoclinic lattice (P2/c space group) occurring in the twinned lamellae form (Fig. 1). Basing on the examinations of thin foils in the transmission electron microscope it was found out that the structure of the investigated Si_3N_4 nitride tool ceramics is the β-Si_3N_4 phase. Moreover, it was found out that the size of the significant portion of the β-Si_3N_4 phase particles is smaller than 500 nm, which unequivocally classifies the investigated ceramics to the fine-grained materials (Fig. 2).

a)



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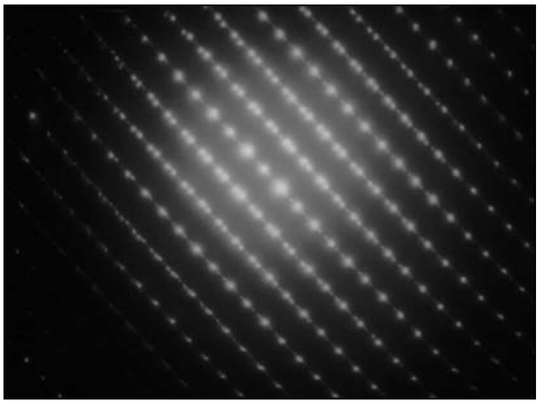

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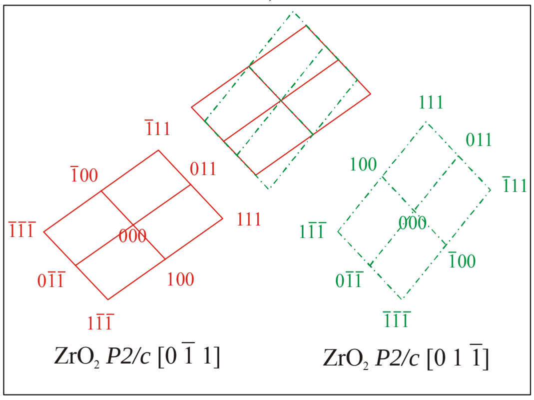


Fig. 1. Structure of Al_2O_3+ZrO_2 substrate: thin foil structure parallel to the layer surface (TEM): a) light field, b) diffraction pattern from the area as in figure a, c) solution of the diffraction patterns from figure b.

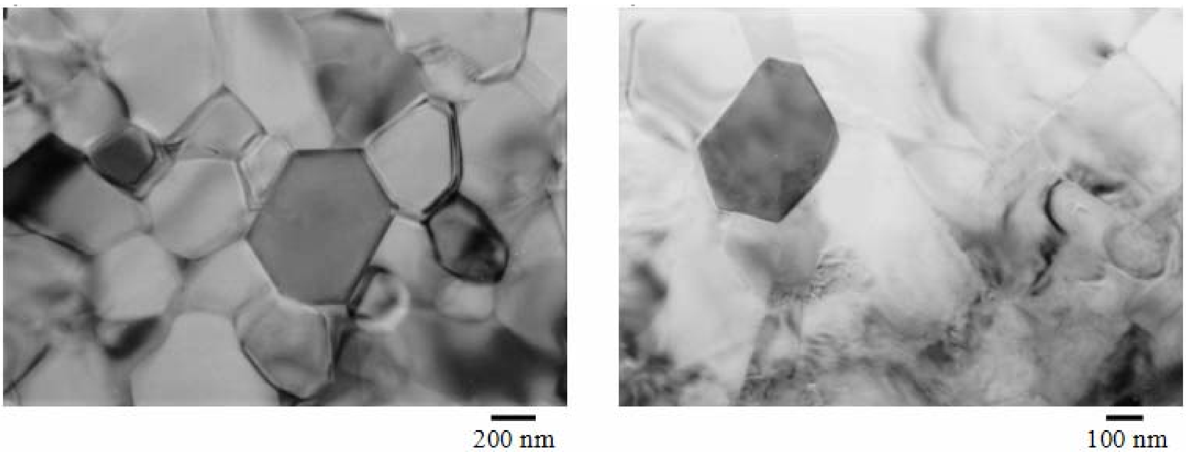


Fig. 2. Structure of tool nitride ceramics Si_3N_4; thin foil structure parallel to the layer surface (TEM)

The fractographic examinations carried out that give grounds to state that the coatings were deposited uniformly onto the investigated substrate materials and that they are characteristic of the depending on the coating type employed, and that the particular layers adhere tightly to themselves and to the substrate (Fig. 3a). Examinations of the chemical compositions of the coatings carried out using the X-ray energy dispersive spectrograph EDS confirm presence of the relevant elements in the deposited coatings and their layers (Fig. 3b-3d).

Topography observations of the investigated PVD coatings reveal their inhomogeneity connected with occurrences of multiple drop shaped micro-particles on coating surface, which is connected with the nature of the employed coating deposition PVD process. Sizes of the micro-particles are diversified and range from several tenths of a micrometer to a dozen or so micrometers (Fig. 4a). The black CVD coating completed with Al_2O_3 has another surface topography. The surface of TiN+Al_2O_3 coating is characteristic of significant inhomogeneity, roughness with the sharp upturned particles (Fig. 4b).

a) b) c) d)

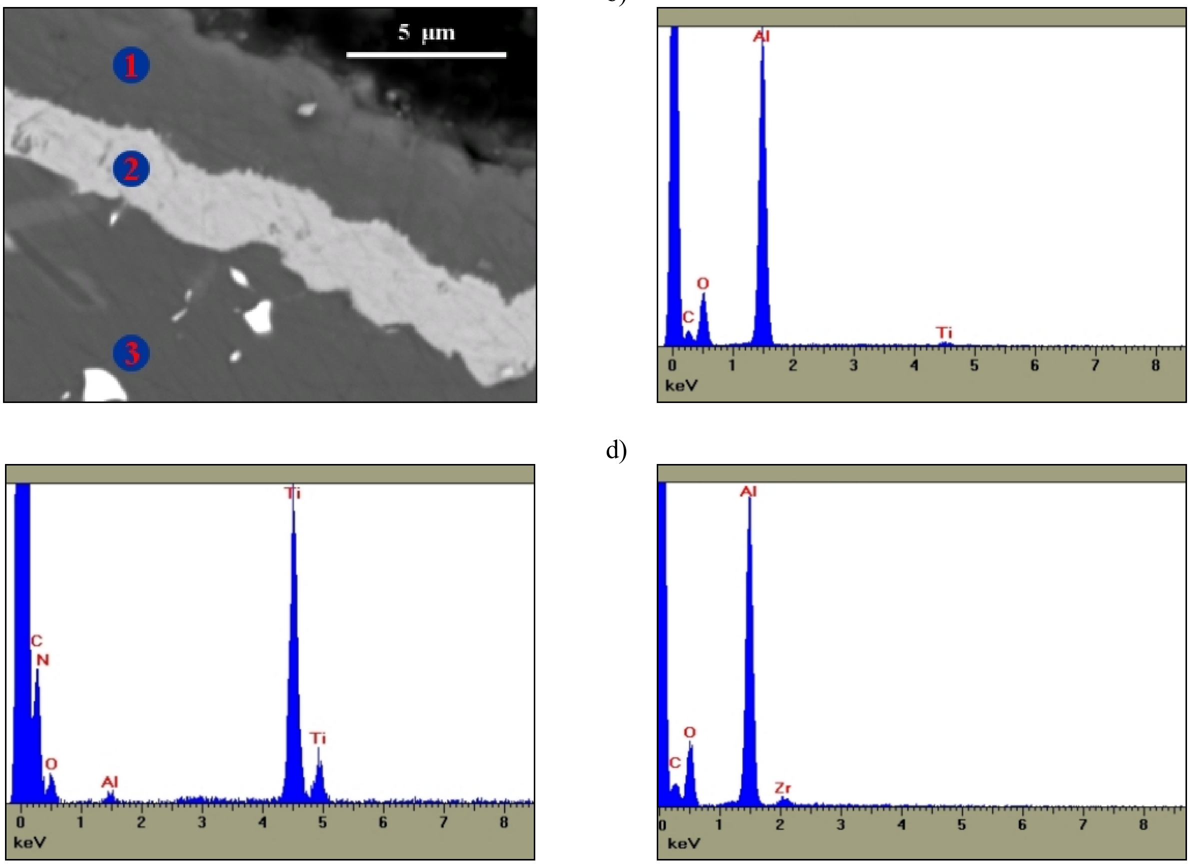


Fig. 3. a) Fracture surface of the $TiN+Al_2O_3$ coating deposited on $Al_2O_3+ZrO_2$ substrate, b) X-ray energy dispersive plot the area 1 as in figure a, c) X-ray energy dispersive plot the area 2 as in figure a, d) X-ray energy dispersive plot the area 3 as in figure a.

a) b)

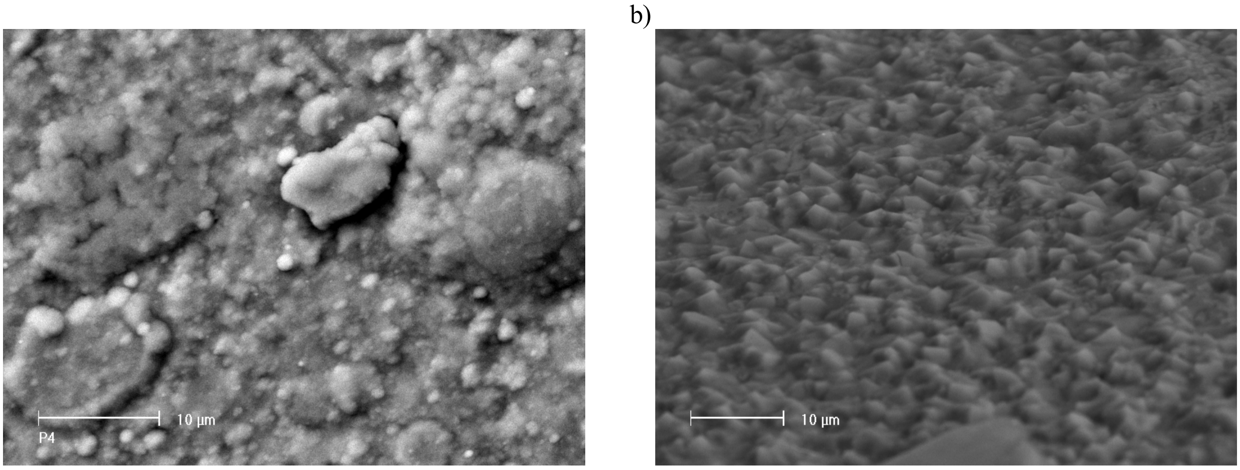
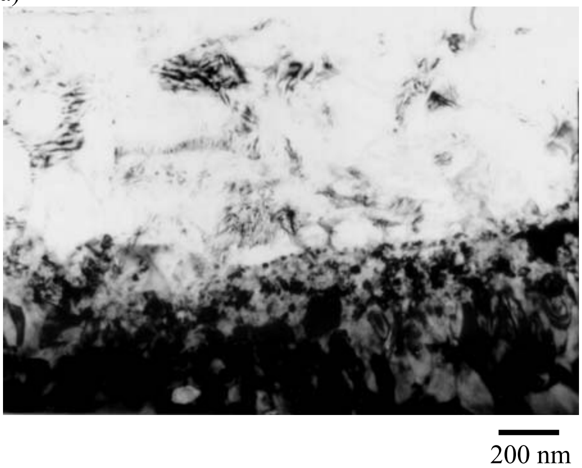


Fig. 4 a) Topography of the a)TiN+TiAlSiN+AlSiTiN and b) $TiN+Al_2O_3$ coatings surface deposited onto the Si_3N_4 nitride ceramics substrate

Examinations of thin foils from the $TiN+Al_2O_3$ (CVD) coating confirm that, according to the original assumptions, coatings containing the TiN and Al_2O_3 type phases were deposited onto the nitride tool ceramics substrate. Examination results of the thin foil from the transverse section of the $TiN+Al_2O_3$ coating indicate that the TiN coating has a columnar structure; whereas, the Al_2O_3 layer – the coarse-grained structure. There is an interface between the TiN and

Al_2O_3 layers, where the fine grains of these phases are found. Occurrences of the scarce fine-grained Al_2O_3 grains with the monoclinic structure were revealed in this zone, unlike the typical structure of the Al_2O_3 phase with the trigonal lattice, which occurs outside of this border area over the entire layer width (Fig. 5).

a)



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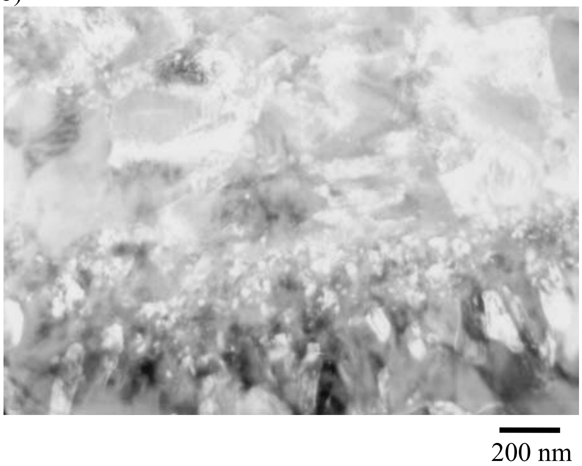

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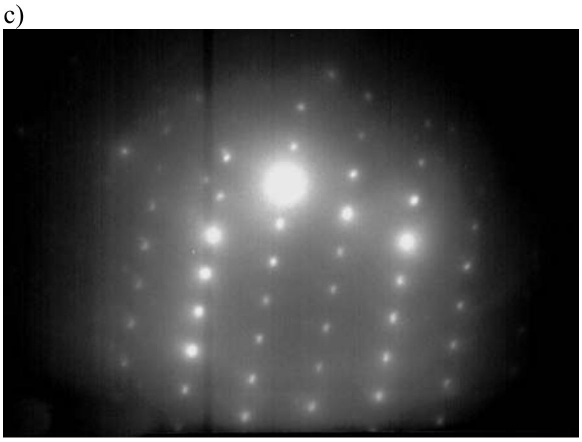

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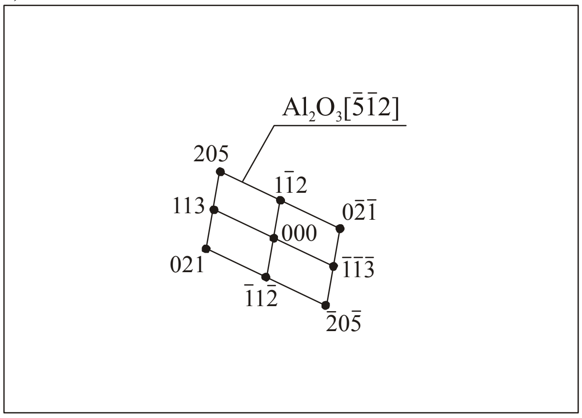
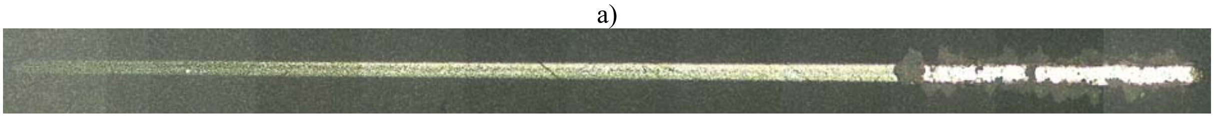


Fig. 5. a) Structure of TiN+Al_2O_3 coating: thin foil from cross section of the layer surface (TEM): b) dark field, c) diffraction pattern from the area as in figure a, d) solution of the diffraction pattern from figure c

a)



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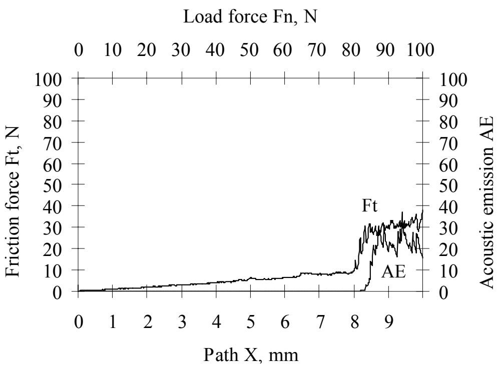

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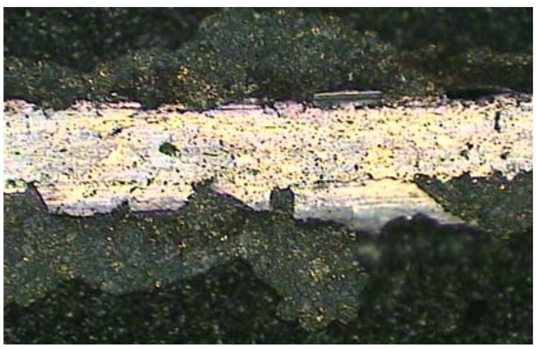

Fig. 6. a) Trace of the scratch test b) diagram of the dependence of acoustic emission (AE) and friction force Ft on the load, c) failure by 90.4 N load – for the TiN+Al_2O_3 coating deposited on Si_3N_4 nitride ceramics (magnification 200x)

Table 2
Mechanical properties of coated tool ceramics

Substrate	Coatings		Micro-hardness, GPa	Critical load, Lc
	Type	Composition		
Si_3N_4	gradient layer	TiN+TiAlSiN+AlSiTiN	26.7	18
	double layer	$TiN+Al_2O_3$	32.5	83
$Al_2O_3+ZrO_2$	gradient layer	TiN+TiAlSiN+AlSiTiN	21.0	(78_{opt})
	double layer	$TiN+Al_2O_3$	34.1	73

The microhardness tests revealed that the uncoated tool ceramics has hardness equal to 18.5 GPa. Deposition of the PVD and CVD coatings onto the specimens causes the surface layer hardness increase reaching from 21.0 to 34.1 GPa, that is up to 90% more compared to the substrate hardness. The highest hardness of 34.1GPa was observed in case of the $TiN+Al_2O_3$ coating deposited on $Al_2O_3+ZrO_2$ substrate. No dependence was revealed between the substrate hardness and hardness of the deposited surface layer (Table 2).

The critical load values L_c (AE) were determined using the scratch method with the lineraly increasing load („scratch test"), characterising adherence of the investigated PVD and CVD coatings to the investigation ceramics. The critical load was determined as the one corresponding to the acoustic emission AE increase signalling beginning of spalling of the coating. To determine the character of the defect responsible for the acoustic emission increase examinations of scratches developed during the test were carried out on the scanning electron microscope and on the light microscope coupled with the measurement device, determining the critical load L_c value basing on the metallographic examinations. It was found out in case of the TiN+TiAlSiN+AlSiTiN coatings that the highest critical load of L_c = 78 N reveals a coating deposition on the oxide ceramics; whereas the lowest one of L_c = 18 N has a coating deposited on nitride ceramic. The CVD coatings deposited onto the investigated substrates are characterised by good adherence compared to the PVD ones, the critical value L_c ranges from 73 to 83 N (Fig. 6) (Table 2).

Basing on the cutting ability test results, the high abrasion wear resistance was revealed of the $TiN+Al_2O_3$ coating deposited onto the nitride ceramics substrate and of the TiN+TiAlSiN+AlSiTiN coating deposited onto the oxide ceramics, compared to the uncoated cutting inserts. As regards the uncoated cutting inserts, the wear trace width on the tool flank varied from VB=0.22 mm for the oxide ceramics to VB=0.30 mm for the nitride one. The assumed tool flank wear of criterion VB=0.30 mm was reached after 8 minutes of cutting. The smallest tool flank wear width of VB=0.08 mm for the cutting speed of v_c=400m/min was demonstrated by the oxide ceramics with the TiN+TiAlSiN+AlSiTiN coating (Fig. 7) and the nitride ceramics with the $TiN+Al_2O_3$ coating (VB=0.16 mm) (Fig. 8).

a) b)

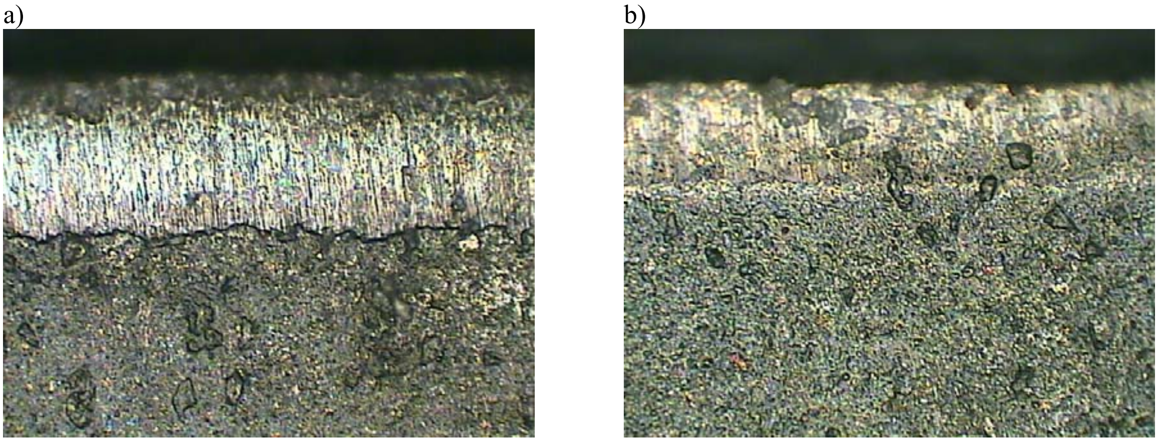

Fig. 7. Wear trace width on the Al_2O_3 ceramics cutting insert flank: a) uncoated, b) covered with the TiN+TiAlSiN+AlSiTiN coating – cutting time 8 min. (magnification 160x)

Measurements of the roughness parameter R_a of the EN-GJL-250 grey cast iron surface after cutting tests with the cutting speed of v_c=400 m/min reveal that the lowest roughness parameter value R_a=1,5 μm is characteristic for the cast iron surface machined with the oxide ceramics coated with TiN+TiAlSiN+AlSiTiN. Measurement results revealed that putting down the TiN+TiAlSiN+AlSiTiN coating onto the oxide ceramics in the PVD process and the $TiN+Al_2O_3$ coating onto the nitride ceramics in the CVD process decreases the roughness parameter of the machined material, compared to cutting with the uncoated tool, which results in quality improvement of the manufactured products (Fig. 9).

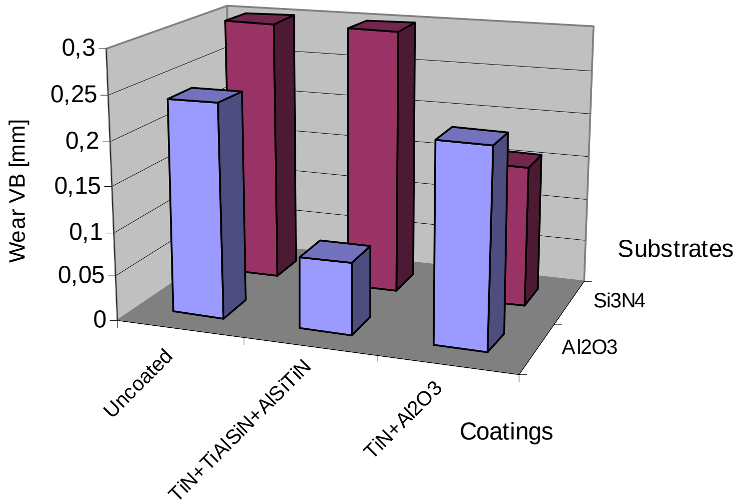

Fig. 8. Values of flank wear width VB after 8 min of cutting the EN-GJL-250 grey cast iron for the nitride and oxide ceramics - coated and uncoated

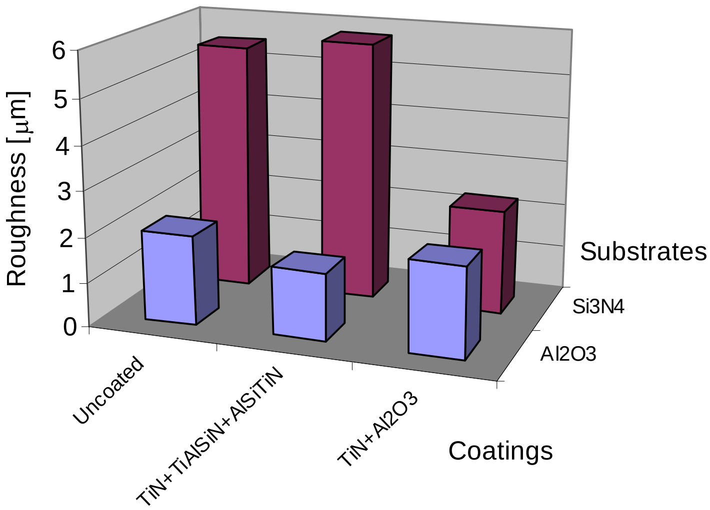

Fig. 9. Roughness parameter R_a values of the EN-GJL-250 grey cast iron surface after cutting tests

4. Summary

All PVD and CVD coatings deposited onto the nitride tool ceramics are characterized by a structure without pores and discontinuities and by tight adherence to themselves and of the entire multilayer coating to the substrate. Basing on the examinations of thin foils in the transmission electron microscope it was found out that the structure of the substrates and coatings is fine-grained - phase particles is smaller than 500 nm

The tool ceramics microhardness grows significantly after deposition of the PVD and CVD coatings. The CVD coatings deposited onto the investigated substrate are characterised by good adherence compared to the PVD ones. The lowest adherence has TiN+TiAlSiN+AlSiTiN coating deposited on nitride ceramics substrate.

It was demonstrated, basing on the technological cutting tests of grey cast iron, that putting down onto the tool ceramics the thin anti-wear coatings in the PVD and CVD processes increases their abrasion wear resistance, which has a direct effect on extending the tool edge life. Basing on the roughness parameter R_a of the machined cast iron surface after the cutting tests, improvement was revealed of the machined material properties, cut with the oxide ceramics with the TiN+TiAlSiN+AlSiTiN coating, and with the nitride ceramics with the TiN+Al_2O_3 coating, compared to material machined with the uncoated tools.

5. Acknowledgements

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