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Comparison of the structure and properties of the PVD and CVD coatings deposited on nitride tool ceramics

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Abstract: Comprehensive structure and properties investigation results of the multilayer, multi-component PVD (Physical Vapour Deposition) and CVD (Chemical Vapour Deposition) coatings developed on the Si_3N_4 nitride tool ceramics substrate are presented in the paper. The detailed results are presented of examinations carried out on the scanning and transmission electron microscopes, as well as of the mechanical properties of the investigated coatings. Machining tests of the nickel alloy were made to analyse in detail the investigated multi-edge cutting inserts.

Keywords: Nitride ceramics; PVD; CVD; Multi-layer coating; Multi-component coatings

1. INTRODUCTION

The dynamic development in the engineering and technology domains gives the reason to increase the requirements posed to sintered tool materials in regard to their mechanical properties and abrasion wear resistance. Functional properties of many products and their elements depend not only on their capability to carry the mechanical loads by the element's entire cross-section from the material used or on its physical and chemical properties, but very often or mostly on its structure and properties of its surface layers. Economical considerations also dictate using the ennobling technologies for surface layer, which ensure the required service properties, making it possible to use simultaneously the possibly inexpensive materials for the element's core, from which lower service properties are usually required. The contemporary technologies of materials forming employed in the machining, plastic forming, casting, and also plastics forming domains call for using more and more efficient tool materials. These requirements pertain mostly to the extension of life and reliability of tools used in machining processes, and also of limiting employment of cutting fluids still commonly used, however degrading the environment considerably. Improvement of the functional properties of tools and reduction of the ecological threats may be accomplished by employing the technology of putting down hard coatings on tools in the CVD processes, and sometimes also in the state-of-the-art PVD processes, mostly by improvement of the tribological contact conditions in the cutting zone and by eliminating the cutting fluids [1-11].

The goal of this work is to comparison of the structure and properties of the PVD and CVD coatings deposited on nitride tool ceramics.

2. EXPERIMENTAL PROCEDURE

The investigations were carried out on the multi-point inserts made from the Si_3N_4 nitride ceramics uncoated and coated in the PVD process with thin coatings. The inserts made from Si_3N_4 were multilayer coated in the PVD process – Cathodic Arc Evaporation (CAE), and were compared next with the commercially available inserts from various manufacturers offering the Al_2O_3+TiN type combination of coatings. Specifications of the investigated materials are presented in Table 1.

Table 1.
Characteristics of the PVD and CVD coatings deposited on the Si_3N_4 nitride ceramics

Coating		Coating thickness, μm	Process type
Type	Composition		
multilayer	TiN+multiTiAlSiN+TiN	4.0	PVD
gradient/multilayer	TiN+TiAlSiN+TiN	2.0	PVD
gradient	TiN+TiAlSiN+AlSiTiN	2.5	PVD
multilayer	TiC+Ti(C,N)+Al_2O_3+TiN	7.8	CVD
two layers	TiN+Al_2O_3	10	CVD
multilayer	TiN+Al_2O_3+TiN	3.8	CVD*
two layers	Al_2O_3+TiN	2.6	CVD*
*commercially available inserts from various manufacturers			

Observations of the investigated coatings' structures were carried out on the transverse fractures on the scanning electron microscope (SEM) Philips XL-3. To obtain the fracture images the Secondary Electrons (SE) and the Back Scattered Electrons (BSE) detection methods were used with the accelerating voltage in the range of 15-20 [kV], maximum magnifications are 10000×.

The diffraction examinations and examinations of thin foils were made on the JEOL JEM 3010UHR transmission electron microscope at the accelerating voltage of 300 kV and maximum magnification 300000x. The diffraction patterns from the transmission electron microscope were solved using the computer program. Thin foils were made in the longitudinal and transverse sections from the 0.2-0.5 mm thick laminae cut from the solid specimens, from which 3 mm O.D. disks were cut out, with the initially ground and levelled surfaces. The lamellae were next mechanically thinned on the Disc Grinder to thickness of about 80 μm and ion polished using the Gatan firm equipment.

The Seifert-FPM XRD 7 X-ray diffractometer, equipped with the texture add-on, was used for evaluation of the coatings' structures. The X-ray radiation was used of the Co Kα cobalt lamp with the 35 kV supply voltage and current of 40 mA. Analysis of the investigated coatings texture was made using the inverse pole figures method. Intensity of the (111), (002), (022) and (113) diffraction lines was analysed – in case of the TiC, TiN and Ti(C,N) phases, and of the (012), (104), (110), (113), (116), (030), and (1010) ones in case of the Al_2O_3 phase.

The microhardness tests of coatings were made on the SHIMADZU DUH 202 ultra microhardness tester. Test conditions were selected so that the required and comparable test results would be obtained for all analyzed coatings. Measurements were made at 0.07 N load, eliminating influence of the substrate 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.

In addition, the tested inserts were used to machine Inconel 718 in the laboratory conditions, with the following parameters: feed rate f = 0.2 mm/rev; depth of cut a_p = 0.5 mm; cutting speed v_c = 250 m/min. The character of the developed failure was evaluated basing on observations on the light microscope.

3. DISCUSSION OF INVESTIGATION RESULTS

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. The comprehensive examinations on the transmission electron microscope (thin foil perpendicular to the layer surface) make it possible to reveal the columnar structure for the TiN layer included in the $TiN+Al_2O_3$. The comprehensive examinations on the transmission electron microscope (thin foil perpendicular to the layer surface) make it possible to reveal such columnar structure for the TiN layer included in the $TiN+Al_2O_3$ coating. These examinations have revealed also that there is an interface between the TiN and Al_2O_3 layers in case of the $TiN+Al_2O_3$ coating, 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. The observed interface zone contributes to good adhesion between these layers, which is also confirmed by high abrasion wear resistance of this coating (Fig. 1).

Evaluating differences of textures of the TiN layers obtained with the PVD and CVD methods one can state that - in the most general understanding - the method of physical deposition from the gaseous phase favours development of the relatively strong texture. It is the {111} + {001} double texture most often; however the {001} component is usually stronger. Also the very weak texture of the carbide and nitrocarbide layers, deposited with the chemical deposition from the gaseous phase method, seems to confirm the rule that this method leads to development of layers with the very weak texture. Crystallisation of the presented PVD coatings is commenced on the preferred plane with the {111} orientation, that is on the plane with the most dense arrangement of atoms, perpendicular to the plasma beam. The strong texture occurs also in case of the TiN+TiAlSiN+TiN, TiN+TiAlSiN+AlSiTiN multi-layer coatings and seems to confirm the epitaxial increase of the consecutive alternating TiN and TiAlSiN layers, being undoubtedly favoured by the isomorphism of the pure titanium nitride and of the secondary TiAlSiN solid solution constituting these coatings, which is consistent with the previous research results (Fig. 2).

Surface layer hardness increase, compared to the uncoated substrate hardness can reach even 100%. Hardness of the investigated coatings systems determines their abrasion wear resistance, which was revealed most clearly for the $TiN+Al_2O_3$ coating - the hardest one from the CVD coatings, adding simultaneously to the decrease of the cutting tools edge wear intensity during machining (Table 2).

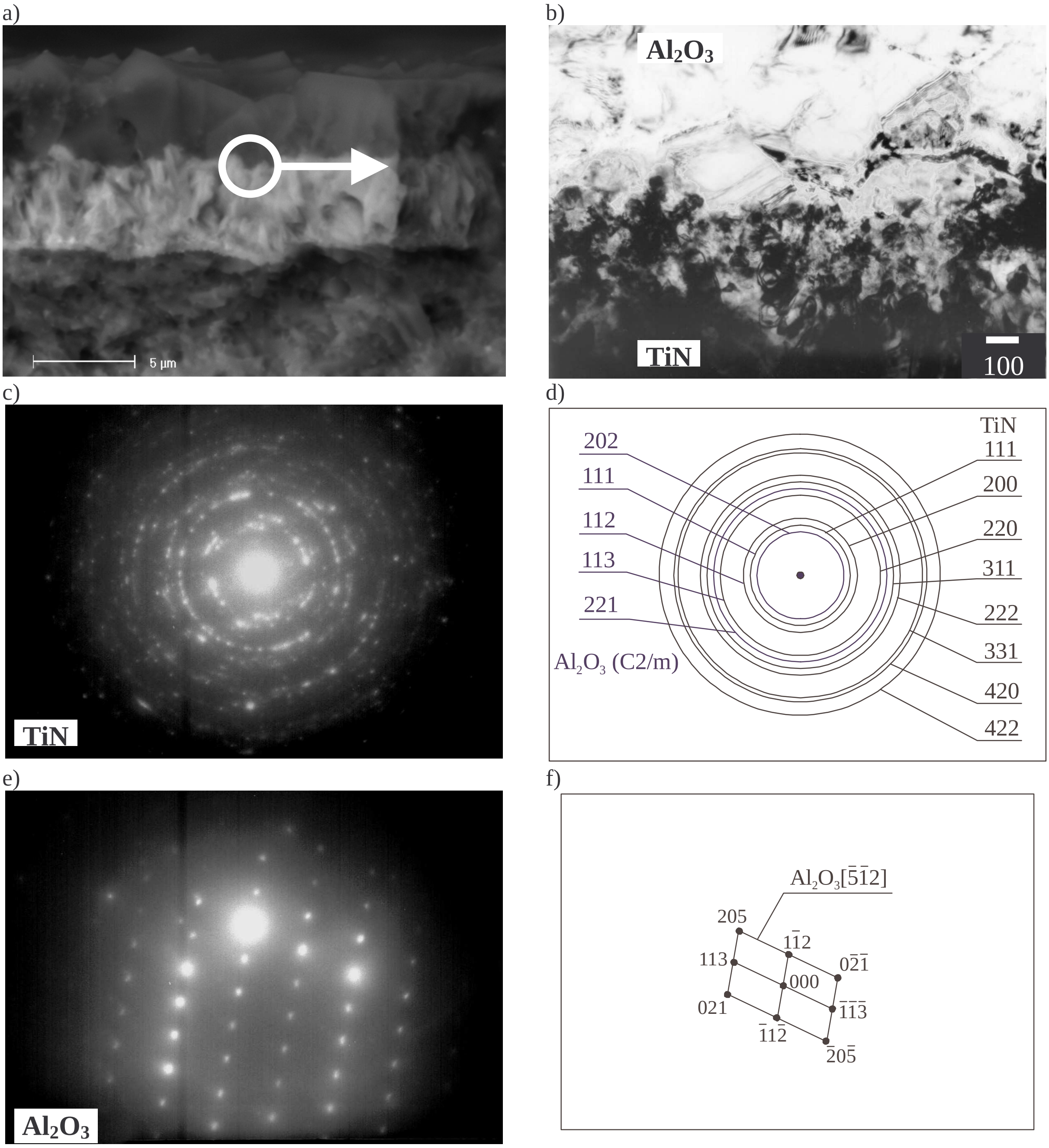
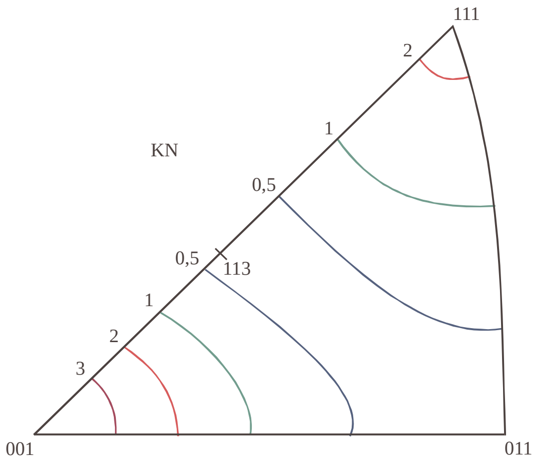


Figure 1. a) Structure of TiN+Al_2O_3 coating, b) thin foil from cross section of the layer surface (TEM) - light field, c) diffraction pattern from the area as in figure b, d) solution of the diffraction pattern from figure c, e) diffraction pattern from the area as in figure b, f) solution of the diffraction pattern from figure e

The CVD coatings, compared to the PVD ones, are characteristic of the very good adherence to the nitride ceramics substrate, which is decided not only by adhesion, but also the diffusion mixing of elements in the interface between the substrate and the coating, and in case of the TiN+Al_2O_3 double-layer coating – mixing of phases in the interface between the particular layers, which – even at the highest load – does not cause total delamination of any coating in the adhesion tests (Table 2).

a)



b)

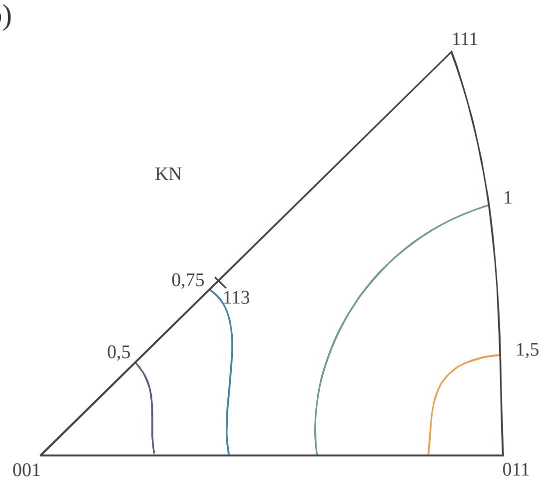
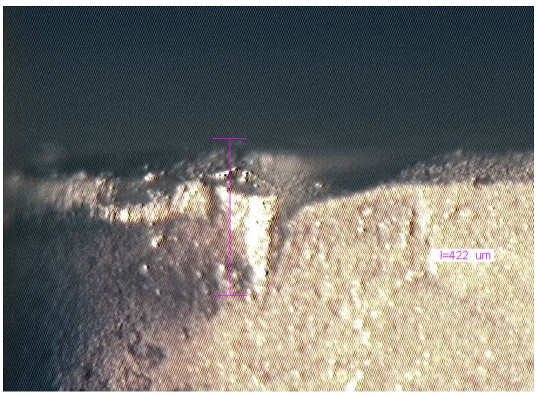


Figure 2. Exemplary inverse for TiN layer representing the distribution of normal to the a) TiN+ TiAlSiN+TiN and b) TiN+Al_2O_3 coating surface in the (001)-(011)-(111) base triangle (KN - normal direction)

Table 2.
Hardness, critical load and tool life of the PVD and CVD coatings deposited on the nitride ceramics

Coatings	Hardness, GPa	Critical load, L_c
–	18.50	–
TiN+multiTiAlSiN+TiN	35.24	22.40
TiN+TiAlSiN+TiN	23.33	21.65
TiN+TiAlSiN+AlSiTiN	26.79	18.30
TiC+Ti(C,N)+Al_2O_3+TiN	29.68	32.17
TiN+Al_2O_3	32.57	83.10
TiN+Al_2O_3+TiN*	24.40	47.78
Al_2O_3+TiN*	26.25	44.60
*commercially available inserts from various manufacturers		

a)



b)

Figure 3. Width of the VB tool flank for nitride ceramics Si_3N_4 with deposited a) TiN+ multiTiAlSiN+TiN coating – after 1.5 minutes and b) TiN+Al_2O_3 coating – after 0.5 minutes of the nickel alloys machining (magnification 50x)

In search of new applications of this selected group of cutting materials an attempt was made of cutting the nickel alloy in the laboratory conditions. These tests reveal that the examined inserts are not up to cutting this type of materials. The only conclusion, drawn from this test is that the PVD coated nitride ceramics - TiN+multiTiAlSiN+TiN and TiN+TiAlSiN+TiN demonstrate better cutting properties, as VB_{max} on these inserts' flanks is significantly smaller that for the selected CVD coated inserts, and especially with Al_2O_3+TiN and TiN+Al_2O_3 (Fig. 3). Regrettably, poor adherence of the PVD coatings determines life of the tool flank. Recapitulating results of these cutting tests one has to stress that there are still chances to substitute the PVD coatings deposited onto the Si_3N_4 nitride ceramics in future for the CVD ones. Undoubtedly, these chances will grow with change of parameters and with modernizing of the PVD processes.

4. SUMMARY

High hardness, good adhesion, and very good abrasion wear resistance - especially noticeable for the nitride ceramics with the Al_2O_3+TiN combination of layers and with the TiN+Al_2O_3 coating, increase the cutting tool flank life and therefore, and also because of the possibility of their use in the pro-ecological dry-cutting processes, without employment of any cutting fluids, make these coatings suitable for many industrial applications on cutting tools.

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