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Investigation of the structure and properties of PVD and CVD coatings deposited on the Si_3N_4 nitride ceramics

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Materials

ABSTRACT

Purpose: Comprehensive structure and properties investigation results of the multilayer, multi-component, gradient PVD and CVD coatings developed on the Si_3N_4 nitride tool ceramics substrate are presented in the paper.
Design/methodology/approach: The detailed results are presented of examinations carried out on the transmission and scanning electron microscopes, as well as of the mechanical properties and tribological tests of the investigated coatings.
Findings: The research carried out proved that depositing the hard, anti wear, multilayer coatings based on the Al_2O_3 and TiN layers onto the Si_3N_4 nitride tool ceramics with the PVD method results in obtaining better functional properties like extension of the cutting tool life, than in case of the uncoated nitride ceramics or coated with the PVD coatings and some CVD ones.
Research limitations/implications: Pro-ecological dry cutting processes without the use of the cutting fluids and in the „Near-Net-Shape" technology.
Originality/value: In the paper the research coatings deposited in the PVD and CVD methods on sintered tool materials carried out in order to improve the tool cutting properties.
Keywords: Tool materials; PVD; CVD; Si_3N_4

1. Introduction

Apart from developing new or modification of the existing manufacturing methods of tool materials, mostly Si_3N_4, the scientific circles and tool manufacturers interest has been growing in particular in improvement of containing these materials with new, hard coatings obtained in the CVD (Chemical Vapour Deposition) – chemical deposition from the gaseous phase - and PVD (Physical Vapour Deposition) – physical deposition from the gaseous phase. Employment of the surface treatment technology for tools made from the ceramic materials with the Si_3N_4 matrix in the PVD processes of physical deposition from the gaseous phase, and in some selected cases also in the chemical CVD process, to obtain coatings with a high wear resistance, makes improvement of properties of these materials possible in dry cutting conditions, among others due to decrease of the coefficient of friction, increase of micro-hardness, improvement of the tribological contact conditions in the tool – machined workpiece contact area, and also because of the protection from the adhesion and diffusion wear. Determining of these properties will make it possible to indicate the optimum application area for both the matrix ceramics materials and also of the investigated coatings, and for evaluating correlation between the laboratory test results and wear determined in the technological cutting tests [1-15].

2. Materials and methods

Experiments were carried out on the multi-point inserts made from the Si_3N_4 nitride ceramics with the multi-layer and multi-component layers deposited in the CVD process with the TiC, Ti(C,N), Al_2O_3 and TiN coating combination, and in the PVD process – cathode arc evaporation – with the TiAlSiN type coatings, which were later compared with the commercial inserts from various manufacturers offering the CVD Al_2O_3+TiN coatings combination type.

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.

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.

Cutting ability of the investigated materials was determined basing on the technological continuous cutting tests of the EN-GJL-250 grey cast iron with the hardness of about 215 HB. The VB = 0.30 mm width of the wear band on the surface of the tool used for machining was the criterion of the cutting edge consumption evaluation. The following parameters were used in the machining capability experiments: feed rate f = 0.2 mm/rev; depth of cut a_p = 2mm; cutting speed v_c = 400m/min.

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 (Fig. 1).

Examinations of thin foils of the TiN+multiTiAlSiN+TiN coating confirm that, according to the original assumptions, coatings containing the TiN type phases were put down onto the Si_3N_4 based tool nitride ceramics substrate. It is not feasible to differentiate these phases from the diffraction point of view, due to isomorphism of the TiN and TiAlSiN phases (Fig. 2).

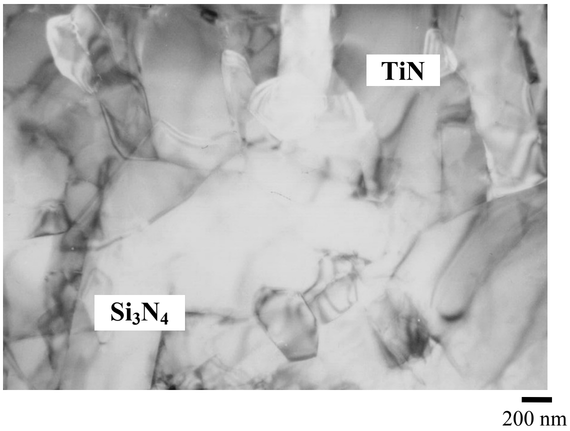

Fig. 1. Structure of TiN+Al_2O_3+TiN+Al_2O_3+TiN coating – deposited on Si_3N_4 substrate + layer TiN; thin foil from cross section of the layer surface (TEM)

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. 3).

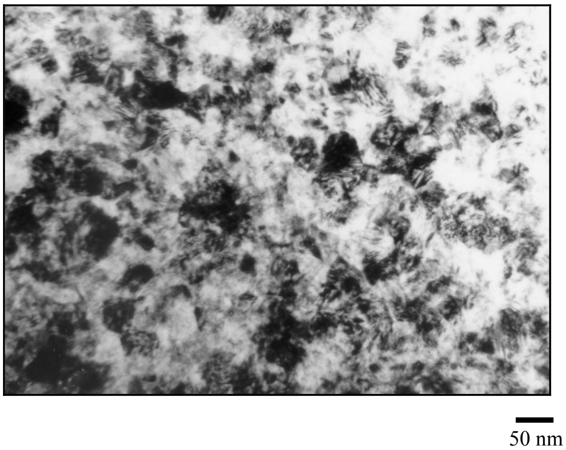

Fig. 2. Structure of TiN+multiTiAlSiN+TiN coating: thin foil structure parallel to the layer surface (TEM)

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

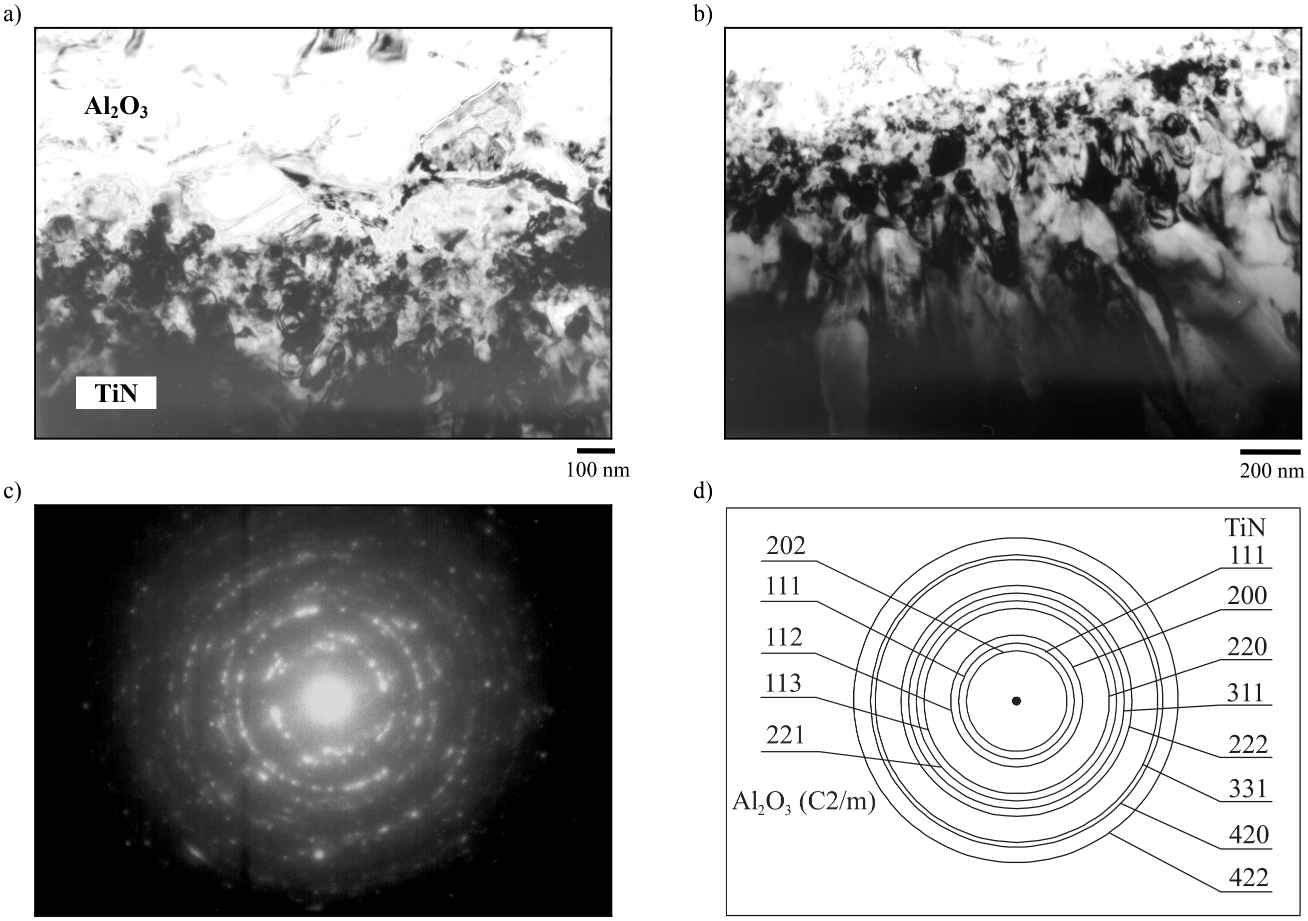

Fig. 3. a) Structure of $TiN+Al_2O_3$ coating: thin foil from cross section of the layer surface (TEM): b) light field, c) diffraction pattern from the area as in figure b, d) solution of the diffraction pattern from figure c

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

Coating			Process type	Hardness, GPa	Critical load L_c, N	Tool live VB, mm
Type	Composition	Thickness, µm				
multi layer	TiN+multiTiAlSiN+TiN	4.0	PVD	35.24	22.40	0.28
gradient/ multilayer	TiN+TiAlSiN+TiN	2.0	PVD	23.33	21.65	0.30
gradient/ multilayer	TiN+TiAlSiN+AlSiTiN	2.5	PVD	26.79	18.30	0.29
two layer	Ti(C,N)+TiN	4.2	CVD	22.25	52.15	0.30
multi layer	$Ti(C,N)+Al_2O_3+TiN$	9.5	CVD	20.11	26.71	0.30
two layer	TiC+TiN	5.4	CVD	19.82	67.24	0.28
multi layer	$TiC+Ti(C,N)+Al_2O_3+TiN$	7.8	CVD	29.68	32.17	0.30
two layer	$TiN+Al_2O_3$	10	CVD	32.57	83.10	0.16
multi layer	$TiN+Al_2O_3+TiN$	3.8	CVD (1)*	24.4	47.78	0.17
two layer	Al_2O_3+TiN	2.6	CVD (2)*	26.25	44.60	0.16
two layer	Al_2O_3+TiN	1.7	CVD (3)*	27.23	26.53	0.19
multi layer	$TiN+Al_2O_3+TiN+Al_2O_3+TiN$	4.5	CVD (4)*	25.22	40.52	0.10

*(1) to (4) commercially available inserts from various manufacturers

simultaneously to the decrease of the cutting tools edge wear intensity during machining. 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 1).

Single point grey cast iron machining tests were carried out to verify correlation between adhesion, hardness of the invesitgated materials, abrasion wear resistance, and cutting ability of the multipoint inserts. All in-service tests are comparative in their nature, i.e., inserts' life was determined basing on the wear band width measurements on the tool flank after cutting during the particular period of time at the identical cutting parameters. Depositing the double layer as well as the multilayer Al_2O_3+TiN and TiN+Al_2O_3 coatings influences significantly the cutting inserts' life. Very good cutting ability of inserts with these coatings, compared to the uncoated nitride ceramics, is connected with their explicit hardness increase, good abrasive wear resistance, and adhesion. Therefore, these basic factors impede the tool flank wear rate for the coated tool materials. Hardness of the investigated coating systems determines their abrasion wear resistance, which was revealed most clearly for the Al_2O_3+TiN coating – the hardest among the CVD ones, contributing at the same time to decreasing the cutting tool edge wear intensity during machining (Table 1).

4. Conclusions

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.

The thermomechanically stable Al_2O_3 layers feature the diffusion barrier between the tool insert and the chip flowing on top of it, which results in improvement of the insert's wear resistance. The TiN layers facilitate chip flow from the machined material by decreasing the friction between the machined workpiece and the multipoint cutting insert. The results herein give the grounds for a statement that depositing of coatings onto the investigated material and the explicit microhardness growth connected with it, impede wear rate of the tool flank of the investigated tool materials.

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