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MULTILAYERED AND COMPOSITE PVD-CVD COATINGS IN CEMENTED CARBIDES MANUFACTURE

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Summary

Carbide cutting tools with wear-resistant coatings deposited by CVD process are widely employed in mechanical engineering to ensure a substantially longer service life of tool systems. However, the relatively high temperature and long time of the process make the substrate decarburise and, as a result, the bend strength and performance characteristics of a tool decrease. The present study suggests the problem of deteriorated strength of CVD-coated carbide tools be solved by the development of a technology that combines arc-PVD and CVD processes to deposit multilayered coatings of titanium and aluminium compounds.

Key words

Supermultilayered coating, combined arc-PVD/CVD technology, tool life

1. Introduction

The recent years saw substantial progress in the improvement of surface properties of tool materials and efficiency and reliability of a wide range of high-speed steel carbide and ceramic cutting tools by various methods of deposition of wear-resistant coatings. With perfected equipment and processes to synthesise the coatings it becomes possible to lay down concepts on the impact of modified surface on a set of properties that define operational performance of cutting tools. Rated among this are hardness, heat resistance, adhesion to a workpiece material, corrosion and oxidation resistance at elevated temperatures, resistance to mechanical and thermal cycling. Service life of cutting tools with wear-resistant coatings depends on

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coating's composition, thickness and number of layers as well as on a synthesis technology.
Present-day technologies are capable of depositing multilayered coatings composed by single, dual and triple compounds of groups IV, V and VI of the periodic system (carbides, nitrides, borides, oxides and their mixtures. Each layer can be made as thin as several nanometres, which allows, in theory, to make it almost defect-free. This way "ideal" properties of a material, in particular, "theoretical strength" could be realised, and multilayered coating should result in the utmost of efficiency and reliability of cutting tools. This makes the tools with coats of this kind instrumental in solving a number of problems for a gamut of cutting operations: improving production rate, precision and quality of machining; reducing the consumption of costly tool materials; implementing environmentally safe cutting with no resort to cutting fluid.

2. A survey of trends in the development of processes to coat carbide tools.

At the present time, chemical (CVD) and physical (PVD) synthesis of coatings are prevalent world-wide in tool-making practice.
CVD processes make use of heterogeneous thermochemical reactions that lead to adsorption and chemosorption with the ensuing coat-comprising compounds being formed in the vapour-gas environment and on the tool's working faces alike /1,2/. The starting materials are gaseous halogens of metals, MeG of which the interaction with other compounds of gas mixtures (H_2, N_2, CH_3, Ar) accounts for the synthesis of a coating.
Properties, structure and quality of a coatings depend on CVD processing variables, among which most important are a time and a temperature of deposition, composition and reactants' content in a vapour-gas medium, as well as a pressure and a flow rate of this. Subject to these variables are structure, phase composition, presence of defects in a coatings, its adherence to a substrate and, therefore, its basic properties. The adhesion strength also strongly depends on the crystallochemical similitude of a coating being deposited and a substrate. As a result, on working faces of a tool a coating is formed that posesses high strength and homogeneity; strong adhesion to substrate; uniform thickness, even on faces of complex shape.
However, CVD processes do not often comply with the environmental safety standards, run at high temperatures (800 °C – 1500 °C) and take long times. The latter results in a brittle η-phase, W_3Co_3C on coating-substrate and grain-

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binder interfaces in carbide, which makes the material's strength decrease for 20% - 30% (1,2) and, in the case of WC-Co substrates, is due to reactions: $(x-2)Me + xWC + yCo \rightarrow (x-2)MeC + W_3Co_3C$.

Besides, characteristic of CVD processes is strong dependence of a coating's quality on a substrate's substructure, because nucleation of condensation sites and growth of coatings display pronounced epitaxy /1,7,8/. In particular, when coatings of TiC-type are being formed on carbide substrates (e.g. WC-Co), condensation nuclei of a coating only arise on cobalt grains because these display a maximum of crystallographic fit. As a consequence, after the coat coalescence completed, straight above carbide grains pores could be formed which proved to be quality-deteriorating defects in coated carbide /7,8/. Therefore in manufacture of these considerable attention is being paid to the grain structure of carbide, which, in particular, is manifested by the employment of fine and ultrafine structures to make pores less likely. In order to check ebrittlement due to η-phase on coat-substrate interfaces, the surface of a substrate is being saturated with carbon prior to coating. Still, there is no adequate solution to the problem of deteriorated strength of carbide inserts after CVD-coating.

PVD processes are essentially generation of matter in an evacuated chamber with reactant gases (N_2, O_2, CH_4, etc.) admitted into it. Particular PVD-technologies differs in the way of matter generation, the extent of ionisation of a vapour-gas stream, the design and operational parameters of installations. Most common in tool-making practice are low-voltage vacuum-arc evaporation systems known as Ion Bond (CIB) systems.

In CIB-processes, matter is generated by the cathode spot of a vacuum-arc of high-current low-voltage discharge within the confines of a stream of vaporised cathode material. The enter of reactant gases (N_2, O_2, CH_4, etc.) into vacuum space are thus assisted by ion bombardment and triggers plasmochemical reactions of the type: $Me^+ + N^+ \rightarrow Me_xN_y$ (in the case of nitrides), which result in coating of working faces of a tool. Arc-evaporation, plasmochemical reactions, ion bombardment and coating processes operate in a vacuum chamber of which the metal case acts as anode.

Characteristic of CIB processes is high reactivity of vaporised media that comprise a highly ionised low-temperature plasma beam (the ionisation degree can be as high as 90% - 95%). A substrate and a deposit undergo intensive bombardment with ions of vaporised metal, which, leads to the surface layers of substrate being partially sputtered and the temperature in a coating zone raised. As a result, atomic mobility on a substrate's surface is intensified, a deposited condensate is further activated, and strong adhesive bonds are formed between a coat and a substrate.

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For CIB processes, of extreme importance are ion-jet density and ion energy during bombardment of a substrate and subsequent coating. The kinetic energy of an ion colliding with a substrate's surface, W_i, is determined by the atomic structure of a substrate, the accelerating voltage U_{IB} applied to this, and the ion charge ratio, eZ, $W_i = W_{I0} + eZ \cdot U_{IB}$, where W_{I0} – energy of ions leaving an evaporator. Subject to exposure time, the energy of ions determines the temperature on a tool's working faces which is of importance for proper thermal activation of a substrate's surface before coating and for the plasmochemical reaction to end in thermodinamically stable, stoichiometric refractory compounds /1,12/.
CIB processes display high production rate, tens of times that of CVD processes which is achieved by the highly ionised beam being accelerated by setting a negative (relative to a chamber's case) potential on a substrate, or by the stream density and homogeneity being enhanced with plasma-optical magnetic systems. One more advantage is that IB processes are environmentally safe.
However, CIB processes have limitations of their own largely due to a micro-droplet constituent of a vapour-ion beam which is most pronounced for metals with relatively low atomic weight and density (Ti, Al, Cr, a.o.). Micro-droplets constitute a coating's structural defect that is most hazardous when located on a substrate-coating interface or on a coating's surface and thereby deteriorate operational performance of a tool /1,11,12/. Besides, in conventional CIB schemes the processes of thermal activation and cleaning of a tool prior to coating are not separated which, on one hand, may lead to a tool's cutting edges being eroded and on the other, results in severely downgraded surface finish of a coating caused by micro-arcs.
Summarily, both CVD and PVD processes still need further development and perfection. And alternative could also be sought in the development of novel processes that should be incorporating the advantages of both.

3. Development of coated carbide tools with improved performance.

Two ways of perfecting carbides with wear-resistant coatings have been tried. One was selection of optimum composition and "architecture" of a coating as well as process variables of a deposition technology that incorporate both chemical and physical methods, whilst the other addressed a carbides of which the structure and properties should comply with the optimum coating conditions and thus result in enhanced cutting performance of a carbide-coating composite.

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To make less likely deterioration of a carbides's strength during coating, caused by decarburising, it has been suggested that a barrier layer should first be deposited with an arc-PVD process and only then the main wear-resistant coatings applied by means of specifically designed CVD-processes. Such a strategy resulted in a coating that consists of a thin underlayer of complex composition and a wear-resistant upper layer. The barrier was intended to arrest interdiffusion between a substrate and a coating, and thereby ensuring decarburising, η-phase formation and embrittlement of a substrate. Besides, the CIB (arc-PVD) processes, employed to deposit an underlayer, also provided for a coating/substrate transitional zone with substantially fewer defects. The upper coating was aimed at the utmost of wear resistance.

To make less likely surface defects inherent to CVD coatings, another sequence of operations have been employed. After deposition of CVD layers, these were first treated with a 1.5 keV metal (e.g. Ti^+) plasma beam (ion bombardment) to eliminate stress concentrators (relief peaks, defective layers of a hardmetal and the like), and the CIB-coated.

A further problem to be solved was selection of a coating's structure and "architecture". Properties were reviewed for several refractory compounds that qualify as wear-resistant coatings on cutting tools, on thermodynamic grounds. A concept was posited of a coating as "technological intermediary" between a tool and a machining materials of which the role is to simultaneously improve the wear resistance of the contact areas and lower the wear-inducing thermomechanical stresses in a tool /1/. Proceeding from the above, a single-layer coating does not seem to comply with the requirements of wear resistance and, therefore, the efforts were largely concentrated on the development of many-layered coatings with varied compositions and properties, to reach the utmost of operational performance of hardmetal tools, especially those intended for severe and intermittent machining.

The study has been conducted in two stages. At the first, to verify the above concepts, at issue was improving the wear resistance of conventional grades with various combinations of multi-purpose layers in a multilayered coating, which were synthesised by a combination of arc-PVD and CVD processes. At the second stage hardmetal substrates were specifically designed to fit best for coating, and the "architecture" of a coating comprised many-layered andmulti-layered structures of compounds TiC – TiCN-TiN and TiC-Al_2O_3 with thin (0.15 μm – 0.2 μm) interlayers of TiN and TiC (CVD processes) and on the base Ti-Al-N compounds (arc-PVD processes). To synthesise multi-

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layered composite coatings, novel equipment and technologies have been designed.

Under study were the properties of conventional carbides grades (T5K10, BK6) with wear-resistant multi-layered coatings TiN – (TiC, TiCN, TiN) (arc PVD/CVD) and (TiC, TiCN, TiN) – TiN (CVD/arc PVD). Tested parameters comprise structure and phase composition, bend strength and its variance, operational performance in longitudinal turning of steel 45 HB 200. Following are some results of the first stage.

A carbide's strength is of most importance in assessing the ability of a tool's contact areas to resist micro-and-macro failure. Simple bend tests were carried out on a 5 mm × 5 mm × 35 mm bars with a coating on the side exposed to tensile stresses. This kind of test was considered as closest to actual stress state in an operating tool and allowing to disclose structural effects.

The results were processed in terms of Veibull theory of brittle fracture based on Veibull-Gnedenko assymptotic distribution of empirical data (Table 1).

Table 1.

BEND STRENGTH (σ_b) AND STRENGTH UNIFORMITY FACTOR (m) FOR 5%TiC – 85%WC – 10%Co (T5K10) HARDMETAL WITH MULTILAYERED COATINGS

Material	T5K10*	T5K10 – (TiC, TiCN, TiN) (CVD)	T5K10 – TiN (PVD)	T5K10 – TiN (PVD)- (TiC, TiCN, TiN) (CVD)	T5K10 – (TiC, TiCN, TiN) (CVD) – TiN (PVD)
σ_b (in MPa)	1320	1100	1330	1330	1150
m	3.8	3.6	4.8	4.8	4.0

As seen in Table 1, combined arc-PVD/CVD coating has left the strength of a carbide substrate almost intact and even imparted substantial uniformity to its empirical values. The most uniform in this respect were specimens with coats of TiN (arc PVD) – (TiC, TiCN, TiN) (CVD) and TiN (arc PVD) that displayed the highest value of Veibull factor m = 4.8 whilst the sequence (TiC, TiCN, TiN) (CVD) – TiN (PVD) resulted in a somewhat lower value.

The results appear consistent with the above suggested surface remedial action of CIB beams with energies of up to 1.5 KeV.

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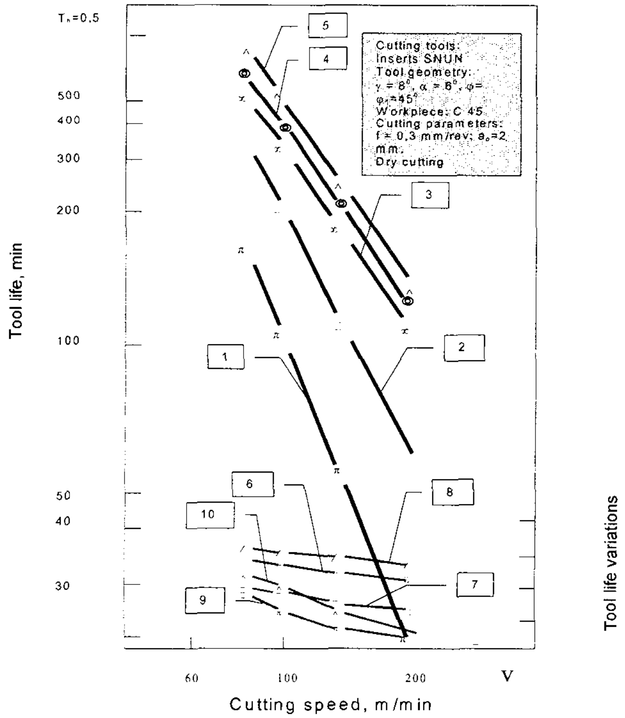
Cutting performance of hardmetals with complex multilayered coats has been tested by longitudinal turning of 45 HB 200 steel blanks of a 160 mm diameter and 400 mm long. The reliability of tests was secured by statistical processing of the results and employment of semifinish cutting, to reduce stochastic spread in values of tool life (Fig. 1).



1,6 – T5K10; 2,7 – T5K10-TiN (arc-PVD); 3,8 – T5K10-(TiC,TiCN,TiN)(CVD); 4,9 – T5K10 – TiN (arc-PVD)-(TiC,TiCN,TiN)(CVD); 5,10 – T5K10 – (TiN,TiCN,TiN)(CVD)- TiN (arc-PVD)

Figure 1. Tool life values of carbide with various combinations of coatings

The T5K10 inserts with TiN (PVD) – (TiC, TiCN, TiN) (CVD) and (TiC, TiCN, TiN) (CVD) – TiN (PVD) coatings (4 and 5 in Fig. 1) display only a slightly improved service life whilst a spread of its values (7 and 8 in Fig. 1) is substantially reduced. The highest stability is imparted to the cutting performance by TiN (PVD) - (TiC, TiCN, TiN) (CVD) coatings with an CIB synthesised TiN interlayer. The tool life data are consistent with those of

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strength tests (Table 1). A composite coating of TiN (PVD) – (TiC, TiCN, TiN) (CVD) also ensured the highest stability of strength.
At the second stage, the substrates were carbide grades specifically adapted for wear resistant coating.
The properties of a substrate grade BK8HC used in the study are shown in Table 2.

Table 2.

TYPICAL PROPERTIES OF BK8HC CARBIDES

HRA	F (HRA), %	He, kA/m	F (He), %	σ_b, MPa	F(σ_b), %	W, wt%	Porosity
90.5	0.12	14.2	2.47	2165	5.2	16.5	A – 0.04

The other substrate was a titanium-tantalum carbide grade BT-141 of GUP VNIITS make, intended for ISO P30-P40 application.
The objective of multilayer coating was considered as follows. Subject to the features of a deposition process, either on a substrate or on an underlying layer crystals are being nucleated, e.g. TiC crystals on a hardmetal surface. The crystal lattice of TiC in a certain position is easy to match the lattice of cobalt and almost incompatible with WC lattice. Therefore, the nucleation and epitaxical growth of TiC crystals should largely occur on a cobalt binder surrounding WC grains.
The values of TiC lattice size, a TiC and residual stresses are a function of a deposit's thickness and substrate's composition. The studies have shown that a TiC had been growing up to 4 nm and no further increase occurred when the growth of a layer proceeded. The latter, however, was accompanied by directional growth of TiC crystals, normal to a substrate's surface. This tendency was found more pronounced for TiN layers. Columnar structures thus formed are uncalled for, because of the low wear resistance.
Several methods have been developed to prevent columnar growth. Most effective proved intermittent deposition of *ultrathin (100 nm – 1000 nm)* layers of compounds that are close to a main layer in terms of composition but differ in a lattice size for over 20%. As a result, columnar crystals stopped growing whilst nucleation of condensation sites and growth of a layer started anew. In selecting the interlayers, provision was made to enhance the overall wear

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resistance of a multilayered coating with compressive residual stress being created in a layer.

Special attention has been paid to the perfection of equipment for CIB coating and elimination of the above mentioned drawbacks inherent to vacuum-arc coating process /9, 10/. The schematic drawing of developed arc-PVD installation is shown in figure 2.

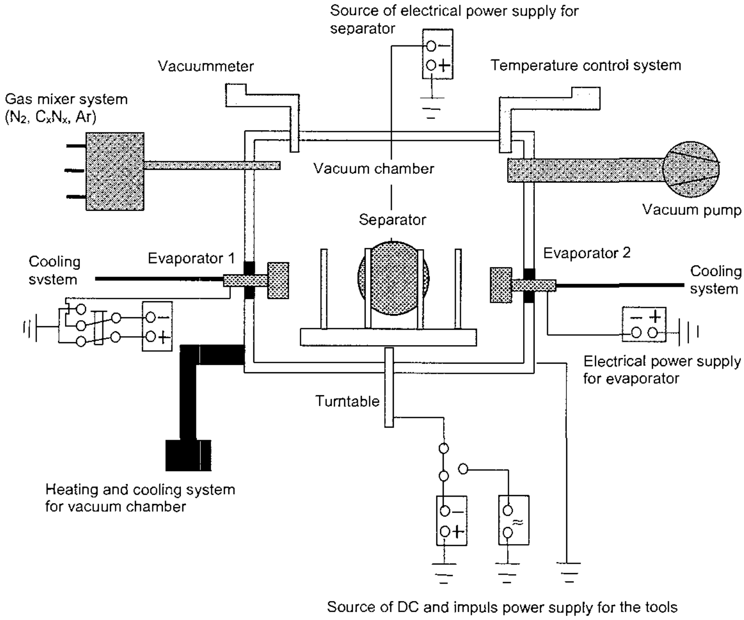

Figure 2. Schematic drawing of a vacuum-arc unit for combined ion plasma surface processing (CIPP) of cutting tools

The installation is equipped with a device to inject an electronic constituent into a chamber which allows to separate cleaning and thermal activation operations and also to select optimum variables of these, irrespective of a charge size in a chamber /12/. The enhanced gas ionising also promotes the coating. The installation's equipment includes a high-voltage pulse source of ions, dynamic gas mixer and a device to separate the droplet constituent of a ion's flow.

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The droplet separator's (Fig. 3) role is manifold. A droplet constituent can be separated by deflecting the charged particles with a strong magnetic field /10/.

The separator can also act as an accelerator for plasma beam, a source of electrons for thermal activation of a tool and a source of high-energy ions of a metal (e.g. titanium) to ensure efficient modifying treatment of a carbide's surface. Thus the installation's design provides for suppression of microareas, feeding gas mixtures of highly precise compositions, almost complete elimination of hazardous droplet defects in a coating.
The coatings have been deposited on inserts in the updated ion-vacuum installation (Fig. 2) with three cathodes, two of which were conventional evaporators with an electromagnetically stabilised cathode spot and plasma-optically focussed plasma beam, and one was equipped with a separation-acceleration system (Fig. 3).

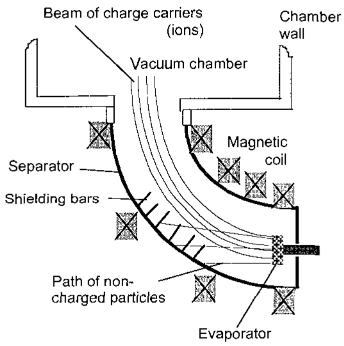

Figure 3. Schematic drawing of a separator devices

Carbide inserts were fed into the unit chamber on a rotary table intended to move a charge in a plasma gas beam. Residual gas pressure in a chamber was adjusted with a vacuum pumping system and automatic bleed-in control, the temperature of inserts was monitored by a pyrometric system "Smotrich-7" in a working temperature range of 150 °C – 900 °C, a wavelength range of 1.8 μm – 3.8 μm, and with a slight-on factor of 1:500 and measuring time of less than 0.025c.
According to the quantative surface analysis of (Ti, Al)N coatings, the Ti/Al ratio is strongly dependent on the synthesis variables (Table 3) especially on a substrate bias voltage, U_c, a working gas pressure, P_N, and Ti evaporating

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are current, I_{Ti}. In particular, when I_{Ti} rises from 40A to 80A, Ti content falls from 21.3 wt % to 18.4 wt %, and Al content, from 44.1 wt % to 40.4 wt %.

Table 3.

COMPOSITION AND MICROHARDNESS OF (Ti, Al)N COATING AS A FUNCTION OF PROCESS VARIABLES

I_{Ti}, A	P_N, Pa	Uc, V	Ti, wt%	Al, wt%	N, wt%	H^{50}_{μ}, GPa
40	$1x10^{-1}$	75	21.3	44.7	34.0	2.370
80	$1x10^{-1}$	75	38.3	33.0	28.5	2.462
60	$1x10^{-1}$	75	18.4	40.4	26.3	2.473

The charges in the contents of a coating's components have pronouncedly influenced its properties (hardness, composition) with the cathode's composition being unaltered during the synthesis of a coating. In turn, the performance of tools equipped with the coated carbide inserts could also be strongly affected.

The improved CIB processes have been implemented on the developed (Fig. 2 and 3) and in this way a novel technology to manufacture multilayered coatings was created. Shown in Fig. 4 is a structure of a multilayered coating with each layer being as thin as 20 nm. The stratified structure comprises alternating dark (Ti, Al) and light Al-rich layers.

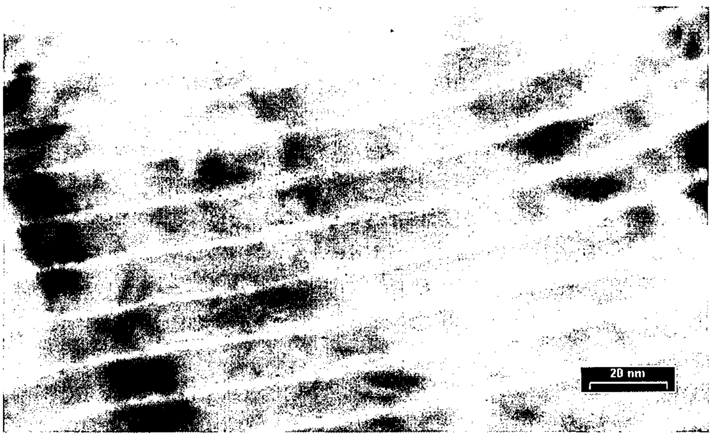

Figure 4. SEM micrograph of (Ti, Al)N multilayered coat (courtesy of A. Panskov)

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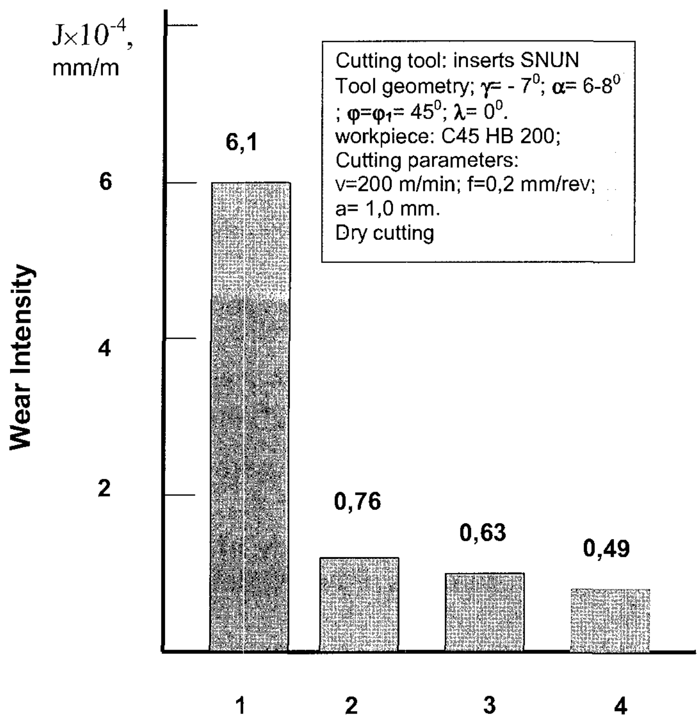
To certify carbide inserts with multilayered coatings of compositions deposited by a novel integrated arc PVD-CVD process, the cutting performance was tested. Under study were many-layered coatings (TiC, TiCN, TiN), CVD; a multilayered coatings on the base of Ti – C – N system, CVD; a supermultilayered coating based on Ti – C – N (CVD) – Ti – Al – N (arc PVD) combination. The coatings with a thickness of 9 μm – 10 μm were upplied to BK8 HC and BT-141 carbide substrates. The tests were carried out and 600 mm long, with SNUN (ISO 120408) inserts. With a square insert clamped in a holder (GOST 20872-80 and GOST 19058-80), the cutting geometry of a tool was: $\gamma = -10°$; $\alpha = 6° - 8°$; $\phi = \phi_1 = 45°$; $\lambda = 0°$. The results are in part reviewed in Fig. 5 and could serve as a proof of advantages offered by the carbide inserts with the coatings of novel compositions.



1 – control carbide; 2 – (TiC,TiCN,TiN) CVD; 3 –supermult. (TiCN) CVD;
4 – supermult. (TiCN) CVD - (TiAlN) - arc-PVD

Figure 5. Wear intensity for varies coated carbide turning tools.

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4. Conclusion:

Novel compositions have been elaborated for multilayered coatings synthesised by a combination of CVD and arc PVD deposition processes. The tools equipped with carbide inserts having the supermultilayered coatings not only displayed improved cutting performance but also were highly reliable and, therefore, can be recommended for application in high speed automated operations, severe cutting conditions, interrupted cutting and, in a number of conditions, for cutting of hardmachining materials.

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