THE STUDY OF TWO TYPES OF PVD COATINGS ON THE SUBSTRATE MADE BY POWDER METALLURGY

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Abstract
This study investigates the properties and cutting performance with thin layers applied by two PVD technologies. PVD methods ARC and LARC were used for the deposition of thin coatings onto tools prepared by powder metallurgy (PM). Advanced types of layers - multilayer AlTiCrN and nanocomposite type of nc-AlTiN/Si_3N_4 layer - were analyzed by standard techniques for surface status and quality assessment – roughness, hardness, layer thickness, chemical composition by GDOES, tribological properties at room and elevated temperature. Durability testing of the cutting tools was carried out according to the standard ISO 3685-1999. The nanocompositenc-AlTiN/Si_3N_4 layer had lower roughness when compared to monolayer AlTiCrN which leads to higher hardness and better layer quality. The HV0.5 hardness values reached nearly 26 GPa. The results showed a two to three times longer durability of the cutting tools in comparison with equivalent uncoated PM and traditional materials. The deposited coatings contributed to the improvement of their durability.

Keywords: cutting tool, PVD layer, roughness, hardness, GDOES, Pin-on-Disc, durability

1 Introduction

Thin films of transition metal nitrides have been widely used in many engineering applications especially due to their high hardness, chemical inertness and excellent wear resistance. Among them, the properties and the applications of TiN coatings have been studied extensively. The main disadvantage of TiN is its limited oxidation resistance (approximately 450–500°C). The addition of other elements such as Al, Cr, Si, etc. increases the oxidation resistance of TiN [1,2]. Recently, from the perspective of environmental conservation, dry machining without the use of cutting fluids has been developed and also cutting speeds have been increased to improve cutting efficiency, causing increased temperatures of the cutting edge [3]. TiAlN coatings have been developed for engineering applications as an alternative to TiN since 1986 [4]. Accordingly, materials that can replace TiAlN are required. In the attempt of adding Cr, research shows that a slight addition of Cr to AlTiN results in excellent performance in the cutting of hardened steels [5]. The cutting tools must be made of material harder than the material which is to be cut, and the tool must be able to withstand the heat generated in the metal-cutting process. To produce quality parts, a cutting tool must have three characteristics: hardness and strength at high temperatures, toughness and wear resistance. So development in the area of cutting tools is focused upon tool surface modification by advanced PVD technologies that are continually improved, and they are generally environmentally friendly because they do not need to use harmful chemical agents and gases. The unique advantage of advanced coatings of [Ti, Al1-xCrx]N and nc-AlTiN/Si_3N_4 types is in their exceptional properties, such as: very high oxidation resistance (above 900°C) with a high hardness of 38-50 GPa [6,7,8]. They are thermodynamically stable materials, also from the point of view of granularity – grain growth does not occur even at temperatures above 1000°C. Grain boundaries act as an effective barrier against defect propagation, and high hardness of these materials is determined in this manner. Layers (Ti, Al, Si)N, that form (Ti,Al)N nanocrystals in a size about 5 nm, are distributed in amorphous Si_3N_4 matrix [9]. Other positive features are the low coefficient of friction, high thermal and low chemical affinity to the machined material [10,11]. Application of these coatings is realizable thanks to new PVD technologies using lateral rotating electrodes, called LARC®–Technology (LAteral Rotating ARC-Cathodes) [12,13]. The techniques described ensure high wear resistance under high - speed machining conditions when cutting tool oxidation wear is dominant. The major cause of the high wear resistance of TiAlN and nc-(Ti1-xAlx)N/a-Si_3N_4 coatings during high - speed machining is the formation of the protective alumina films on the cutting tool surface [14]. Evaluation of some properties of the system thin layer – substrate needs specific methods and procedures. The most important mechanical properties from the point of view of this application are hardness and the adhesion of thin coating to substrate [15, 16].

2 Experimental procedure

2.1 Substrate and coating

Tools from PM high speed steel were coated by PVD technology with composite thin layers of the thickness approx. 3 μm. The coated specimens, and for comparison non-coated ones as well, were subjected to selected testing analyses. Coatings were deposited by means of a physical vapour deposition (PVD) process. Two techniques for coating were used – the classic ARC method with its four planar electrodes located in the corners of the chamber (Cr, AlTi, Cr, AlTi) on "PLATIT 1000" equipment [17] and progressive LARC® –Technology (LAateral Rotating electrodes) for coating deposition nc-AlTiN/Si_3N_4 (*designated as nACo*). An improvement of this modern technology is based on the rotating cathodes and their lateral position. The deposition unit was equipped with the Lateral Arc Rotating Cathodes (LARC) system [17] with two laterally rotating cathodes. During the process, evaporated metals and metal alloys enter the plasma state to combine with the ionized process gas (nitrogen) and eventually condense on the substrate surface, as part of ceramic compounds. Amorphous and micro-nanocrystallinestructures and layers are developed with optimized thermodynamic and kinetic conditions. Spinoidal decomposition allows building TiNnanocrystalline structures dispersed in a Si_3N_4 amorphous matrix, with a typical crystallite size of about 10 nm [12].

2.2 Laboratory tests

The roughness of the coating surfaces was analyzed by Atomic Force Microscopy (AFM, Dimension icon by Veeco Instruments) from the statistically significant compilations, the so-called flat sectors of the coated surface samples. The dimensions of the particles were also defined by the linear method on the surface of the layers and documented in 2D and 3D modes.

Hardness was measured by the microhardness tester LECO LM 700 AT with a load of 0.5 N, and the results were statistically treated using 50 values from each sample. The coated and, for comparison, also the non-coated specimens were subjected to the selected testing analyses. *Pin-on-disc tests* were used to evaluate wear resistance of the deposited coating. Fundamental information about behaviour of the thin layer-substrate system was obtained by analysis of the trace after the pin movement and the extent of the track deterioration. The tests were realized at room and elevated temperature (400°C) with a constant load of 5 N on the pin (WC ball was used as a pin) with a sliding speed of 4 cms^{-1}. The result was a graphical record of the coefficient of the friction course during ca 10 000 cycles.

2.3 The GDOES analyse

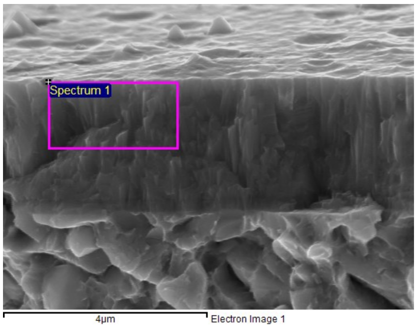
The GDOES method (Glow Discharge Optical Emission Spectroscopy, GDS–750) was used for qualitative and quantitative determination of metallic and non-metallic elements through a cross section of applied layers up to a depth of 0.1 mm of the investigated material. From the concentration profile it is possible to approximately deduct the layer thickness as well. Conversion accuracy of the measurement time axis to the concentration profile axis is given by determination of the exact removal rate of individual calibration standards. Because the measured structures are heterogeneous and multi-component, mistakes in these recalculations are to be expected and it is necessary to proceed from one case to the next [18].

2.4 Cutting test

The tests were realized according to the standard STN ISO 3685-1999 „Durability testing of turning tools with one cutting edge“. A durability test of the cutting tools with applied coatings was carried out under pilot conditions by the so-called long-time cutting test. Cutting blades of the type SPUN 120504 were prepared for long-term durability testing according the standard ISO 3685-1999. The steel (ISO 683/1-87) was used as machining material with tensile strength Rm ≤ 700 MPa [19]. The cutting process is focused upon the area of cutting tool edge contact with machined material in the cutting zone. The properties of machined surface in the cutting zone depend on conditions that influenced the formation of the machined surfaces. The most important conditions are: tool geometry, stress in the machined material, shape of the tip, cutting temperature, etc. [19].

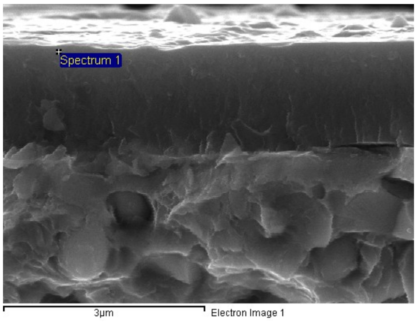
3 Results and discussion

One of the reasons for selecting the material in question and its subsequent testing is that currently there is not a wide range of applications for thin or nanocomposite PVD coatings for parts and tools produced by powder metallurgy technology. The basic physical properties of coatings include roughness, hardness, thickness, adhesive properties, etc. Surface defects that originate in consequence of material defectiveness and its damage, such as an occasional, singular incident and irregular unevenness (scratches, cracks, holes, etc.) are not considered in the course of roughness measurement. Roughness was measured by a profilometer, but for roughness determination in more detail an AFM was used. The morphology of the fracture of coatings is characterized by a dense structure, in some cases there is a columnar structure, **Figs. 1, 2**.



Element	Weight%	Atomic%
N K	*18.49*	*41.48*
Al K	*14.81*	*17.24*
Ti K	*18.53*	*12.16*
Cr K	*48.17*	*29.12*
Totals	**100.00**	

Fig. 1 SEM of the system PM HSS + AlTiCrN and EDS analysis.



Element	Weight%	Atomic%
N K	*33.77*	*58.10*
Al K	*18.32*	*16.36*
Si K	*4.05*	*3.48*
Ti K	*43.85*	*22.06*
Totals	**100.00**	

Fig. 2 SEM of the system PM HSS + nACo and EDS analysis.

The fracture surface of the steel samples was examined and the deposited coatings show a sharp transition zone between the substrate and the coating. The morphology of the surface was documented and evaluated by the AFM method for the "mirror" treated surface before and after application of the surfaces in question. Roughness Ra analyzed on the surface of the substrate before deposition was 4.69nm. By application of the multilayeredAlTiCrN the roughness defined at the temperature 20°C rose to 21.3 nm and the nanocomposite layer nACo only to a value of 12.9 nm. Realizing the pin-on-disc test at a temperature of 400°C enabled the evaluation of the coating roughness after the samples remained at this temperature, while only a negligible drop in roughness was recorded, in **Table 1**.

Table 1 Investigated parameters for the evaluation of coatings attained from the tests realized at room temperature and at 400°C (after the test).

Investigated system	Ra [nm]	Rq [nm]	HV0.5 [GPa]	μ_{aver}
PM HSS	4.69 ± 1.11	6.49 ± 1.52	11.8 ± 1.4	-
PM HSS + AlTiCrN (20°C)	21.30 ± 4.61	37.74 ± 9.15	23.5 ± 4.1	0.74 ± 0.07
PM HSS + AlTiCrN (400°C)	19.77 ± 4.78	35.74 ± 8.15	22.1 ± 0.5	1.27 ± 0.14
PM HSS + nACo (20°C)	12.90 ± 1.64	23.00 ± 3.08	25.9 ± 0.7	0.73 ± 0.08
PM HSS + nACo (400°C)	11.22 ± 2.04	18.10 ± 3.72	21.6 ± 0.3	0.99 ± 0.10

Ra – means arithmetical divergence of profile, Rq – means quadratic value of profile roughness,
HV-hardness at a load of 0.5 N, μ_{aver}– average coefficient of friction

In comparing the two coating technologies applied on surface substrate it can be said that by the advanced technology of LARC with the deposition nanocomposite layer nACo a lower roughness was attained by 40 % compared to the monolayer AlTiCrN. It is related to the composition of the amorphous and nanocrystal constituent of the layer and the LARC technology method. Results of tribological tests showed that elevated temperature did not significantly influence the roughness and hardness when compared to the results obtained at room temperature, in **Table 1**. A decrease of roughness ensures a higher hardness that is necessary for operating cutting tools. For thin layers the hardness is defined as „resistance against penetration of outside subjects“. The standard process of tool wear is given by abrasion. It is a reason that high hardness is a basic parameter of abrasive resistant layers. The hardness of base PM HSS material was 11.8 GPa after heat treatment, the surface modification by coating increased the hardness for both systems to 23.5 and 25.9 GPa. Through a roughness decrease of the nACo coating applied by LARC technology, a ~ 10% (25.9 GPa) increase of hardness was reached in comparison with values measured out on AlTiCrN coating. The values of friction coefficient for individual testing specimens correspond to the layer deposition mode. Lower values of the coefficient of friction were attained for nACo layer applied by LARC technology at both temperatures (0.73 – 0.99) in comparison with values for the AlTiCrN layer (0.74 – 1.27), see **Table 1**. Increasing the friction coefficient at a high temperature has a connection with oxidation causing surface changes. Apart from the coefficient of friction the character of tribological traces and proportion of its failure is also an important evaluative criterion of coating wear degree. It is possible from the character of traces to state that after the pin-on disc test, even at an increased temperature (400°C), no significant uncovering of substrate occurred, in other words no adhesive failure of the coating-substrate system took place. The oxidized particles as residues of the contacting materials appeared on the outside borders of the traces, **Figs. 3 and 4**.

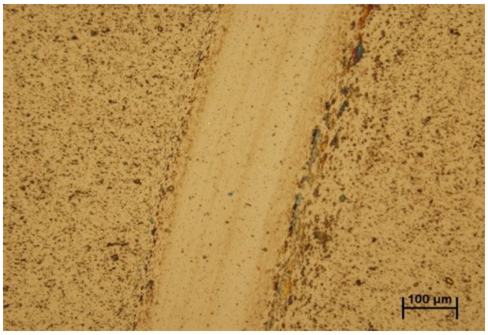


Fig. 3 LOM image of tribological trace in the AlTiCrN layer after the test realized at 400°C.

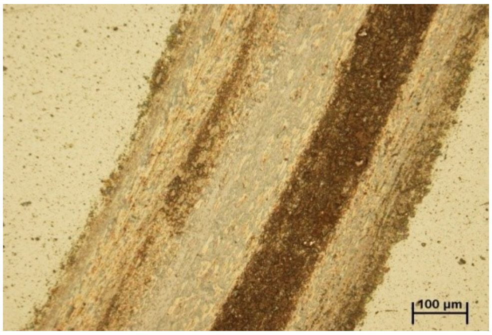


Fig. 4 Fig. 4 LOM image of tribological trace in the nACo layer after the test realized at 400°C.

A gradual uncovering of individual layers according to their configuration from the surface occurred by the acting of the Al_2O_3 ball. It corresponds to the good adhesion properties of coating and substrate. Illustrated in **Fig. 5** are surfaces of the monolayer AlTiCrN and nanocomposite layer nACo in AFM 3D profile, where different morphologies of the surfaces are apparent.

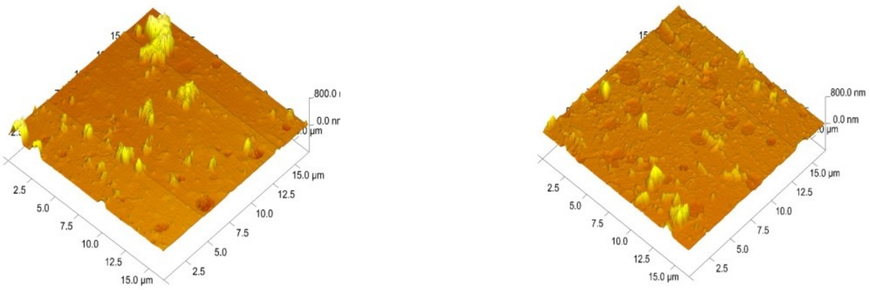


Fig. 5 AFM 3D profile of surface morphology of AlTiCrN and nACo layers.

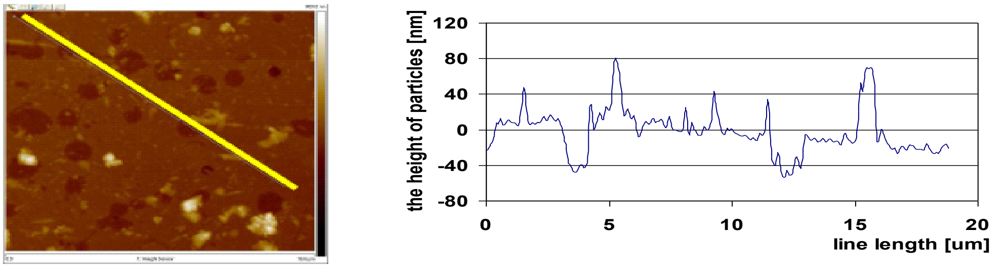


Fig. 6 AFM 2D profile of the surface and graphical record of undulations of the nACo layer.

The linear method AFM evaluated the surface size of particles from several sectors. The height of some microparticles for AlTiCrN coating tested at 20°C was ca 210 nm and after testing at 400°C some particles reached the height of 250-350 nm. Much smaller particles were detected on the nACo coating, max. 80 nm after testing at 20°C and the height of particles after testing at 400°C was max. 105 nm. Presented for illustration in **Fig. 6** is a fragment from the surface of the nanocomposite layer nACo, from which the size of the particles by linear methodwas established. **Figures 7 and 8** present graphical records of the fractions of selected elements in atomic percentages from the coating surface towards the substrate.

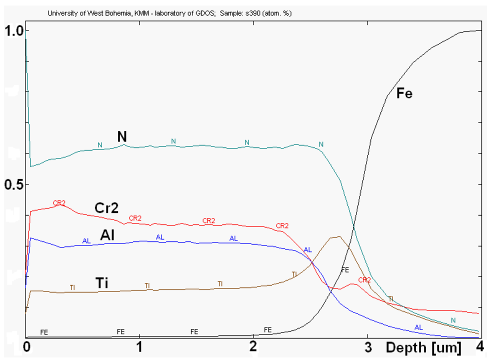


Fig. 7 Depth concentration profile of the surface PM HSS + AlTiCrN system

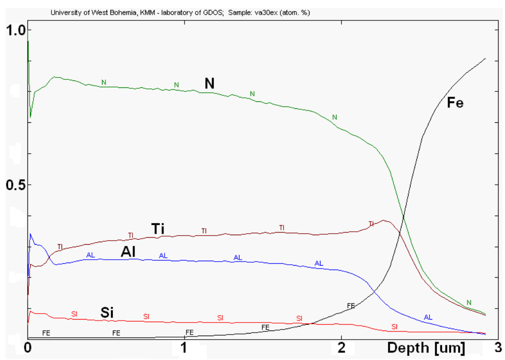
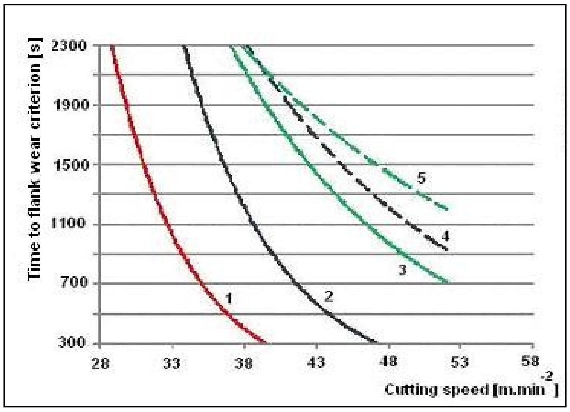


Fig. 8 Depth concentration profile of the surface PM HSS + nACo system

The lines characterize the concentration of analyzed elements in applied thickness of layers. Records can also be used for approximate determination of applied layer thickness. Regarding the fact that the system thin layer - substrate has at present the widest application for cutting tools, a technological test of cutting tips durability seems to be essential for examining the properties of the system. This test detects the influence of individual mechanical and physical properties of the whole system. Substantiation of this test lies in the possibility of utilizing cutting tools from high speed steel for particular service in which exploitation of cutting tools such as cutting ceramics and hard materials is difficult or disadvantageous from financial point of view [19]. Wear of the tool tip during machining is caused by individual wear mechanisms acting on the individual tool areas and by accompanying thermal degradation. The tool edge or tip, respectively, is blunting during cutting, it means that it loses its original geometric shape. This wear occurs by several mechanisms, e.g. by abrasion of material segments of the tool on the face and on the back. The tool is worn out on the face by contact with the chip, on the back by contact with area of the cut [20]. **Figure 9** represents the results of technological cutting tests by radial turning according ISO 3685-1999 (time to flank wear criterion vs. cutting speed). The flank wear criterion on the main dorsal area was determined at the value of vb = 0.6 mm. For completing the test, substrates (equivalent) made by melt metallurgy and powder metallurgy technology without coating were also compared. Based on evaluation of the plots, it is visible that the time of the wear criterion was prolonged with increasing cutting speed in favour of new coatings applied by both technologies. Results of the test point to the better behaviour, or the longer service life for testing tools from powder metallurgy production and with the applied coatings.



1 - MM (melt metallurgy)
2 - PM HSS
3 - PM HSS
4 - PM HSS + AlTiCrN
5 - PM HSS + nACo

Fig. 9 Results from long-time cutting tests for the cutting tools tested.

Of course there is also the influence of the advanced LARC technology with the deposited nanocomposite layer displaying excellent results in cutting tests. Cutting PM materials with deposited coatings AlTiCrN and nACo attained 2-3-times higher durability in comparison with the equivalents made by powder metallurgy without coatings. The fact that cutting tools made by powder metallurgy are advanced and attractive material is proven in observation that these materials attained durability 3-times higher in the pilot process in comparison with tools made by classical metallurgy and the equivalent chemical composition.

4 Conclusions

Selected characteristics were measured such as roughness, hardness, resistance to wear, service life and microstructure at the systems layer – substrate after applying two types of coatings of separate PVD technologies. Deposition of coatings on a quality-prepared surface of substrate from powder metallurgy production increased the roughness of the surface by almost four times; which is the incidental character of PVD technology. NanocompositenACo coating prepared by advanced LARC – technology had roughness parameters ~ 40% lower than monolayer AlTiCrN. This is connected with the composition of amorphous and nanocrystalline components of the layer and the method of LARC technology. The deposited coatings achieved excellent hardness values. Decrease of roughness assured higher hardness, the hardness value (load 0.5 N) was ca 24 GPa for AlTiCrN layer and ca 26 GPa for nACo layer. Hardness values of the specimens with deposited layers are higher by 100% in comparison with the hardness values of the substrate (ca 12 GPa). Results gained by the pin-on-disc test indicated good adhesion properties of the coatings and minimal differences in the friction coefficient for both layers at room temperature. Keeping the coated samples at 400° C and the subsequent roughness evaluation led to a heightened friction coefficient, which may be explained by the influence of oxidizing effects on the surface changes.

A technological test of radial turning showed that the tested cutting PM materials with AlTiCrN or nACo layers had 2-3-times higher durability than equivalent materials without coatings. Cutting tools made by classical melt metallurgy had durability 3-times lower than equivalent material made by powder metallurgy. Realization of both PVD technologies during coating deposition showed excellent results obtained by the tests in question, while very good results were also attained by application of the nanocomposite layer nACo by LARC technology.

Based on the realized tests and their results, it is possible to notice that coated as well as uncoated cutting tools produced by powder metallurgy start to be promising cutting materials in the area of shaping machining at low cutting speeds, where it is necessary to achieve high dimensional accuracy.

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