CHARACTERIZATION OF THIN PVD COATINGS DEPOSITED ON PM HIGH SPEED STEEL

D. Jakubéczyová, P. Hvizdoš, M. Selecká

Abstract

Two PVD techniques (ARC and LARC) were used for deposition of thin coatings onto cutting tools prepared by powder metallurgy. Advanced types of layers - monolayer 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 temperatures. Durability testing of the cutting tools was carried out according to the standard ISO 3685-1999. The nanocomposite nc-AlTiN/Si_3N_4 layer achieved lower roughness when compared to monolayer AlTiCrN which leads to the achievement of higher hardness and better layer quality. The HV0.5 hardness values were ~ 26 GPa. The results showed two to three times longer durability of cutting tools in comparison with the equivalent uncoated PM and traditional materials. The deposited coatings contributed to the improvement of their durability.

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

INTRODUCTION

Production improvements in the engineering industry are influenced by increasing requirements upon quality, functionality and the durability of cutting tools. The lifetime of a cutting tool markedly influences the price of the final product. 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. Also, the tool (cutting blade) must have a specific geometry, with clearance angles designed so that the cutting edge can contact the workpiece without the rest of the tool dragging on the workpiece surface. The angle of the cutting face is also important, as is the flute width, number of flutes or teeth, and margin size. In order to have a long working life, all of the above must be optimized, plus the speeds and feeds at which the tool is run. To produce quality parts, a cutting tool must have three characteristics:

- hardness — hardness and strength at high temperatures;
- toughness — toughness, so that tools do not chip or fracture;
- wear resistance — having an acceptable tool life before needing to be replaced.

So development in the area of cutting tools is focused on tool surface modification by advanced PVD technologies which are continually being improved, and they are generally environmentally friendly because they do not need to use dangerous chemical agents and gases. This fact stems from the principle of physical evaporation process of material, which is a base of the final coating. The unique advantage of advanced coatings of [Ti, $Al_{1-x}Cr_x$]N and nc-AlTiN/Si_3N_4 types is in their exceptional properties such as: very

Dagmar Jakubéczyová, Pavol Hvizdoš, Marcela Selecká, Institute of Materials Research, Slovak Academy of Sciences, Košice, Slovak Republic

high oxidation resistance (above 900°C) with a high hardness of 38-50 GPa [1-5]. 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 in this way high hardness of these materials is determined. Layers (Ti, Al, Si)N, that form (Ti,Al)N nanocrystals sized about 5 nm, are distributed in amorphous Si_3N_4 matrix [6]. Other positive features are the low coefficient of friction, high thermal and low chemical affinity to the machined material [7-10]. Application of these coatings is realizable thanks to new PVD technologies using lateral rotating electrodes, so called LARC®–Technology (LAteral Rotating ARC-Cathodes) [11,12]. Within the contribution the above-mentioned layers deposited on specimens from high speed steels, which were tested by selected methods, are described. These methods are in correlation and together they can give us information about the quality of applied layers.

EXPERIMENTAL

Two high speed steel grades of S 390 Microclean (*signed S 390*) and Vanadis 30 – Super Clean (*signed VA 30*) produced by powder metallurgy were used as the base materials. Selected material grades belong to the group of high performance high speed steels alloyed with cobalt, with excellent abrasion resistance, good toughness and machinability. Their commercial chemical composition is listed in Table 1.

Samples from these materials were coated by PVD technology with composite thin layers of thickness ca 3 µm. Monolayer AlTiCrN was deposited on S 390 steel specimens by ARC technology (two planar electrodes). The method is based on the material evaporation from electrodes (4-plain electrodes located in the chamber edges) by means of a low-voltage arc.

Tab.1. Commercial chemical composition of PM high speed steels used.

Material	Chemical composition [wt.%]							
	C	Cr	Mo	W	V	Co	Si	Mn
VA 30	1.30	3.99	4.81	6.17	3.1	8.20	0.64	0.24
S 390	1.65	4.70	1.88	10.13	4.75	7.77	0.61	0.29

Nanocomposite layer nc-AlTiN/Si_3N_4 (*designated as nACo*) was applied on VA 30 steel specimens by advanced LARC®–Technology (lateral rotating electrodes). An improvement of this modern technology is based on rotating cathodes and their lateral position. The coated specimens, and for comparison also non-coated ones, were subjected to selected testing analyses. Figures 1 and 2 show the cross-fracture of coating – substrate systems and EDX analysis.

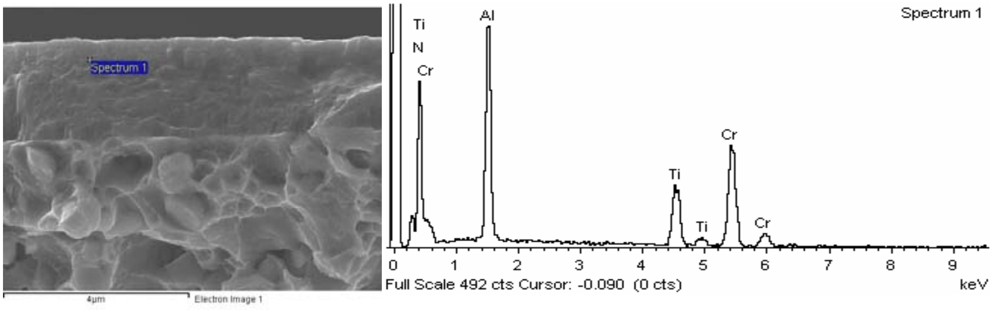

Fig.1. Cross-section of the system S 390 - AlTiCrN and EDS analysis.

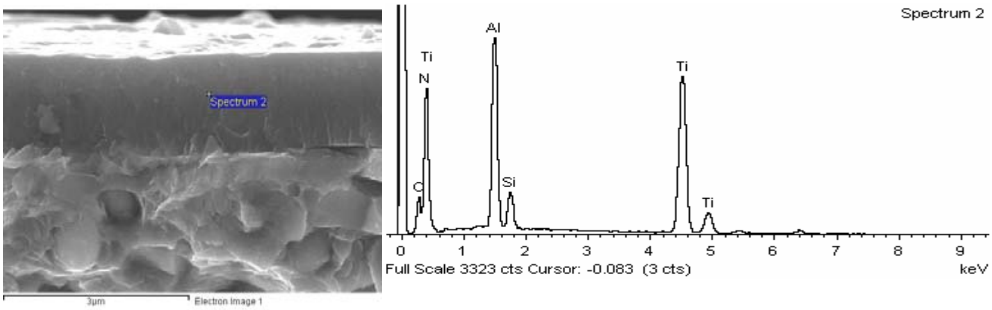

Fig.2. Cross section of the system VA 30 - nACo and EDS analysis.

Roughness of coatings was analyzed by Atomic Force Microscopy (AFM, Dimension icon by Veeco Instruments) with the so-called linear methods 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 from many data. The coated, and for comparison the non-coated specimens as well, were subjected to selected testing analyses.

For evaluation of wear resistance of deposited coating respectively, pin-on-disc tests were realized. These tests rank among the most frequently used methods for the determination of tribological properties of the thin layer-substrate system. The testing includes phenomena related with friction and wear between two moving surfaces (loaded pin and the investigated system) and its result is the trace after wear [13]. 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 both room and elevated temperature (400°C) with a load of 5 N on the pin (a Al_2O_3 ball was used as a pin) with a sliding speed of 4 cms^{-1}. The result was a graphic record of coefficient of friction course during ca 10 000 cycles.

A suitable additional method for acquiring ideas about layer configuration and abrasion resistance is the Calotest. A rotating steel ball of a diameter of ca 25 mm forms a trace on the specimen surface. This trace called calotte allows one to calculate the layer thickness and also to monitor relevant changes and failure of the layer at transitions between layers up to the substrate. A method description of more detail is in [14].

For a qualitative and quantitative determination of metallic and non-metallic elements through cross section of applied layers, GDOES (Glow Discharge Optical

Emission Spectroscopy) analysis was used, realized on optical emitting spectrometer with GDS–750 glow discharge. Its output is tabular values and a vertical concentration profile allowing a graphic record of the concentration changes of selected elements up to the depth of 0.1 mm of the investigated material. From the concentration profile it is possible to deduct approximately 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. There is no universal way for elimination of these inaccuracies, and it is needed to proceed from one case to another [15]. Therefore it is necessary to correct the increased or lowered signal intensity according to the principle of respective calibration. Nearby emission lines have a cardinal importance in the case of quantitative measurement of a chemical composition. Selection of individual wave-lengths for analytical purposes is subjected to some steps, which are listed in the methods described in [16].

The application of thin and hard layers is most exercised on cutting blades and also on tools. The tests were realized according to the standard STN ISO 3685-1999 „Durability testing of turning tools with one cutting edge“. The 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. This steel corresponds to the 41 2050.1 steel (according STN 41 2050) with tensile strength $Rm \leq 700$ MPa [17].

Also in this case, roughness is one of the important characteristics of surface profile. Roughness represents a height of undulations from an unbroken and ideal smoothed surface and it is also a result of the used tool and related parameters. The cutting process is focused upon the area of cutting tool edge contact with machined material in cutting zone. The properties of the machined surface in the cutting zone depend on conditions which influenced the formation of machined surfaces. The most important conditions are: tool geometry, stress in machined material, tip formation, cutting temperature, etc. [17,18]. The stress generated by a cutting implement is directly proportional to the force with which it is applied, and inversely proportional to the area of contact. Hence, the smaller the area (i.e., the sharper the cutting implement), the less force is needed to cut something.

RESULTS AND DISCUSSION

Basic physical properties of coatings include roughness, hardness, thickness, adhesive properties, etc. In the case of PVD layers` deposition, the final roughness is influenced by the quality of tool machining as well as by the applied coating. Surface defects that originate as a 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. The sources of increased roughness of layers are macroparticles that originate during the technological process of coating. Coating roughness increases cutting forces, therefore thermal and mechanical stress of the cutting section of tools occur. Roughness is possible to measure normally by profilometer, but for roughness determination in more detail the AFM is suitable.

It is generally known that the application of PVD layers is realized on perfectly processed base material, in our case Ra ~10-20 nm, and deposition causes an increase of roughness because of macroparticle existence which is an accompanying phenomenon of this surface modification. Roughness values of deposited layers were increased by 5 – 7 times in comparison with plain substrate.

The roughness - values of unevenness of height - of monolayer AlTiCrN and nanocomposite nACo coatings after having realized pin-on-disc tests at temperatures of 20 and 400°C was deducted from graphical records of objective curves. The pin-on-disc test is based on continuous monitoring of the friction coefficient at simultaneous wearing of the investigated surface. The main variable parameters influencing friction and wear are speed of rotation and applied load. Comparison of PVD technologies used and of the influence of elevated temperature for both coating types showed that LARC method caused ~ 30% decrease of nACo coating roughness in comparison with monolayer AlTiCrN. Results of tribological tests showed that the elevated temperature did not significantly influence the roughness and hardness when compared to the results obtained at room temperature (see Table 2). A decrease of roughness ensures a higher hardness which is necessary for operating cutting tools. For thin layers, the hardness is defined as „resistance against penetration of outside objects“. The standard process of tool wear is given by abrasion. It is the reason that high hardness is a basic parameter of abrasive resistant layers. The hardness of base PM HSS materials (S 390, VA 30) 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. By way of a roughness decrease of nACo coating applied by LARC technology, a ~ 10% (25.9 GPa) increase of hardness was reached in comparison with values measured on the AlTiCrN coating. The values of the coefficient of friction for individual testing specimens correspond to the layer deposition mode. Slightly lower values of the coefficient of friction were attained for the nACo layer applied by LARC technology at both temperatures (0.728 – 0.987) in comparison with values for the AlTiCrN layer (0.736 – 1.274), see Table.2.

Tab.2. Investigated parameters for coatings` evaluation attained from the tests realized at room temperature (before the test) and at 400°C (after the test). Ra – mean 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.

Investigated system	Ra [nm]	Rq [nm]	HV0.5 [GPa]	μ_{aver}
S 390	4.0	5.69	11.8 ± 1.4	-
S 390 + AlTiCrN	28.7	56.1	23.5 ± 4.1	0.736 ± 0.068
S 390 + AlTiCrN (400)	28.0	59.0	22.1 ± 0.5	1.274 ± 0.141
VA 30	4.6	6.3	12.4 ± 0.6	-
VA 30 + nACo	20.4	37.9	25.9 ± 0.7	0.728 ± 0.081
VA 30 + nACo (400)	18.5	23.5	21.6 ± 0.3	0.987 ± 0.099

Apart from coefficient of friction, the character of tribological trace and proportion of its failure is also an important evaluative criterion of coating wear degree. From the character of traces it is possible to state that after a pin-on disc test even at increased temperature (400°C) there did not occur any significant uncovering of substrate, in other words no adhesive failure of the system coating-substrate took place.

The oxidized particles as residues of the contacting materials appeared on the outside borders of traces (Figs.3 and 4). 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 base material.

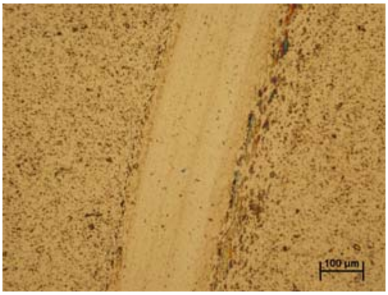

Fig.3. Character of tribological trace in AlTiCrN layer after the test realised at 400°C.

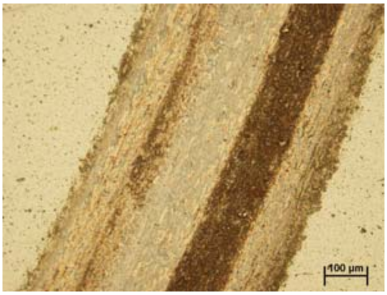

Fig.4. Charakter of tribological trace in nACo layer after the test realised at 400°C.

For illustration, there were selected some spots in the range of applied coatings which present scatter of macroparticles` dimension. 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, see Figs.5-8. Much smaller particles were detected on nACo coating, max. 80 nm after testing at 20°C and the height of particles after testing at 400°C was max. 105 nm. Figures 5 - 8 represent surface fragments from both layer types on the substrate S 390 and VA 30 obtained by AFM.

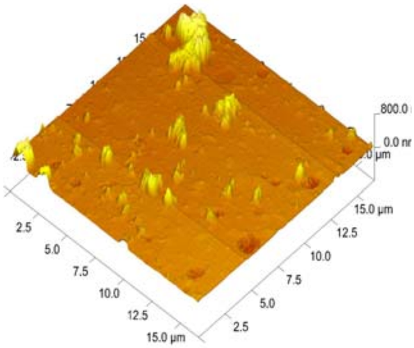

Fig.5a. Surface morphology of AlTiCrN (S 390) layer at 20°C, 3D profile.

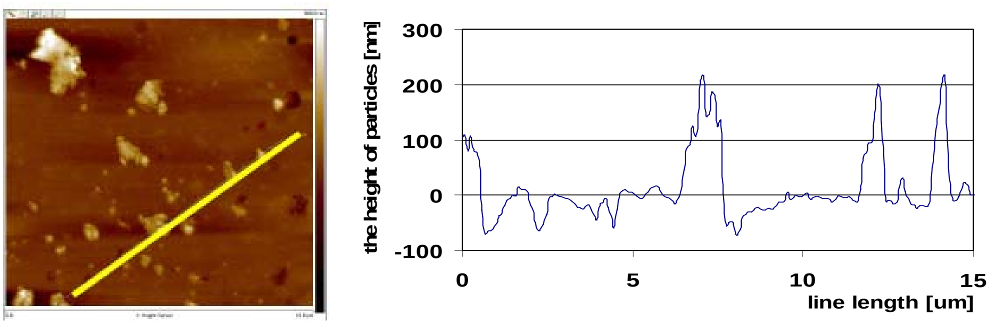

Fig.5b. Layer surface in 2D profile and graphical record of undulations of AlTiCrN layer at 20°C, the height of some particles attained h ~ 210 nm.

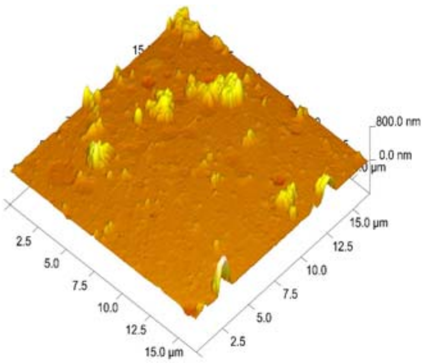

Fig.6a. Surface morphology of AlTiCrN (S 390) layer at 400°C, 3D profile.

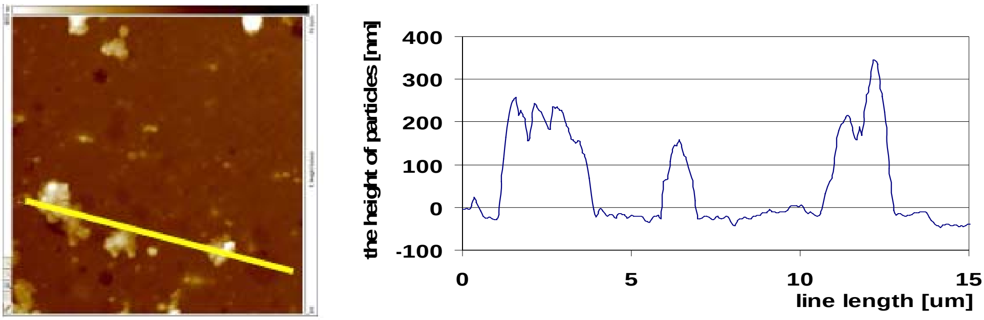

Fig.6b. Layer surface in 2D profile and graphical record of undulations of AlTiCrN layer at 400°C, the height of some particles attained h ~ 250-350 nm.

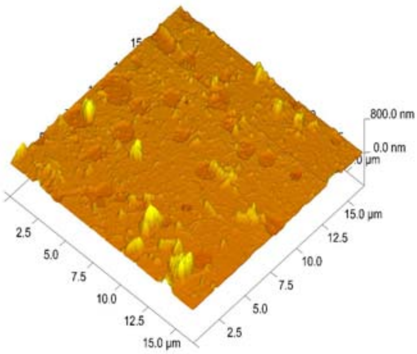

Fig.7a. Surface morphology of nACo (VA 30) layer at 20°C, 3D profile.

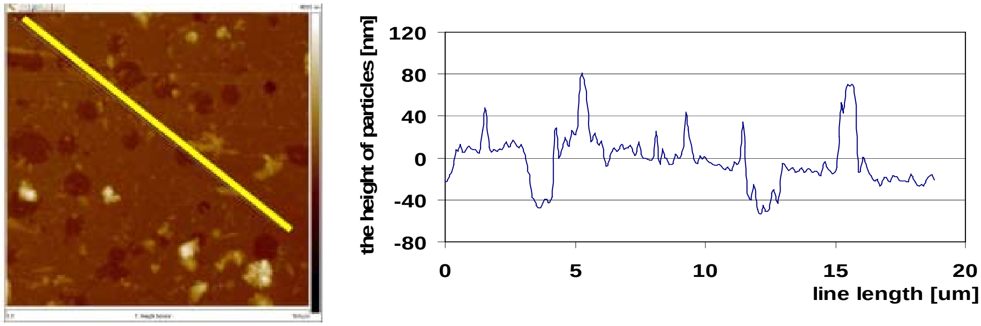

Fig.7b. Layer surface in 2D profile and graphical record of undulations of nACo layer at 20°C, the height of some particles attained h ~ 80 nm.

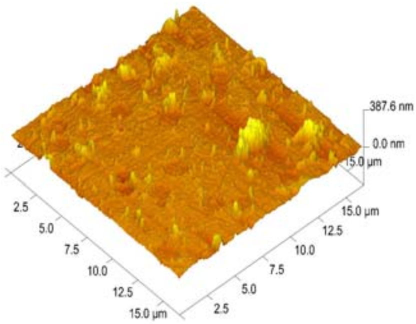

Fig.8a. Surface morphology of nACo (VA 30) layer at 400°C, 3D profile.

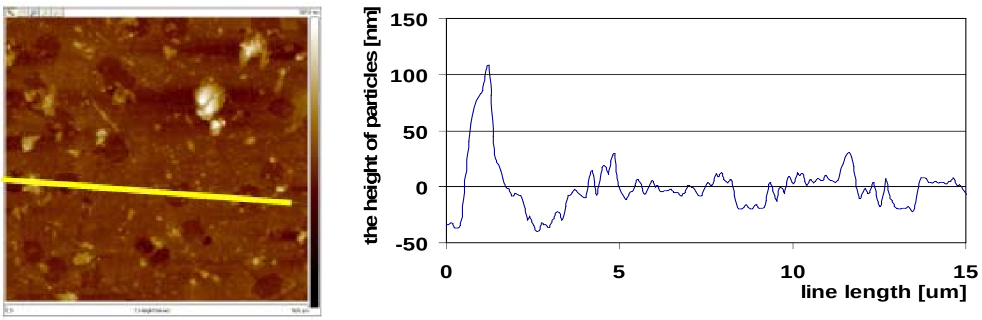

Fig.8b. Layer surface in 2D profile and graphical record of undulations of nACo layer at 400°C, the height of some particles attained h ~ 105 nm.

Measurement of chemical composition by GDOES method is a relative measurement; hence data are measured in one period and at one calibration. Final recalculated values are consistent for a measured set of specimens and it is possible to compare measured values. The method allows determining not only chemical composition of electrically conductive volume materials, but also coatings and thin layers of metal nitrides, etc. The output is tabular values and depth concentration profiles which allow depicting graphically the changes of element content up to the depth of 0.1 mm. These values give information necessary for identification of material and surface processes [15].

Figures 9a and 10a present graphic records of the fractions of selected elements in atomic percentages from the coating surface towards base material. The lines characterize a concentration of analyzed elements in applied thickness of layers. Records can also be used for approximate determination of applied layer thickness. These results were corroborated by testing using the Calotest method (Figs. 9b and 10b). The calottes had a regular shape on the tested specimens, the layers were not broken, in the vicinity of the trace there were no cracks and the substrate was worn in the direction of the ball movement. From these facts we may also deduct good layer abrasion resistance.

Regarding the fact that the system thin layer - base material has at present the widest application for cutting tools, a technological test of cutting edges/tips durability seems to be essential for examination of 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 services for which exploitation of cutting tools such as cutting ceramics and hard materials is difficult or disadvantageous from a financial point of view [17].

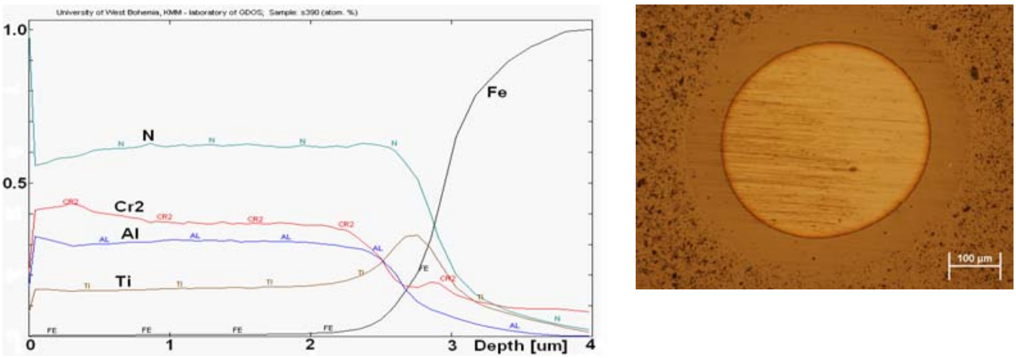

Fig.9. Depth concentration profile (a) and the calotte (b) of the S390-AlTiCrN system.

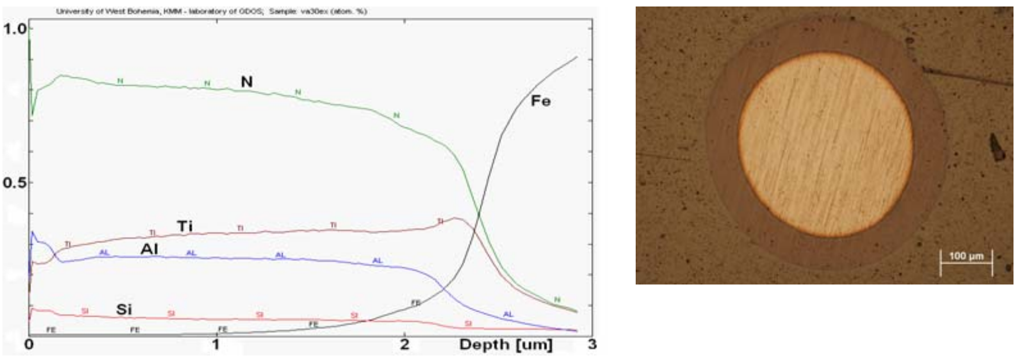

Fig.10. Depth concentration profile (a) and the calotte (b) of the VA 30-nACo system.

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 the area of cut [19].

Figure 11 represents the results of technological cutting tests by radial turning according to ISO 3685-1999 (time to flank wear criterion vs. cutting speed). The flank wear criterion on the main dorsal area was determined at a value of v_b = 0.6 mm. For completing the test, base materials (equivalent) made by melt metallurgy and powder metallurgy technology without coating were also compared. Based on evaluation of 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. 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 proves an observation that these materials attained 3-times higher durability in pilot process in comparison with tools made by classical metallurgy and equivalent chemical composition.

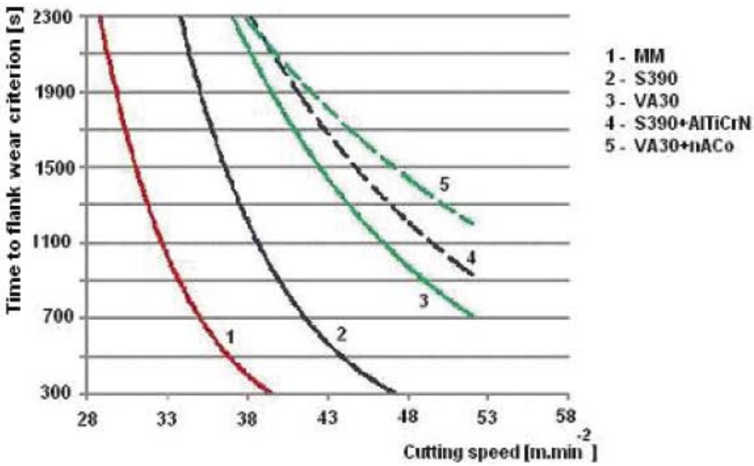

Fig.11. Results from long time cutting tests for tested cutting tools.

CONCLUSIONS

Based on the realized tests and their analyses, it can be concluded:

- Nanocomposite nACo coating prepared by advanced LARC – technology had roughness parameters nearly 30% lower than monolayer AlTiCrN.
- Decrease of roughness assured higher hardness, the hardness value (load 0.5 N) was ca 24 GPa for the AlTiCrN layer and ca 26 GPa for the nACo layer. Hardness values of the specimens with deposited layers are higher by 100% in comparison with the hardness values of the base material (ca 12 GPa).
- The results from the Pin-on-Disc tests showed that the material system VA 30 + nACo (LARC-technology) attained the value 0.728 for friction coefficient for the test at room temperature; this value is lower when comparing it with the S 390 + AlTiCrN material system.
- Calotest and GDOES methods were used for evaluation of configuration, thickness and chemical composition of layers. These methods complemented quantitative evaluation of applied coatings.

- The technological test of radial turning showed that the cutting PM materials tested with AlTiCrN or nACo layers had 2-3-times higher durability than equivalent materials without coatings. Cutting tools made by classical melt metallurgy had 3-times lower durability than equivalent material made by powder metallurgy.

Based on the realized tests and their results, it is possible to notice that coated and 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.

Acknowledgements

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