COMPARATIVE STUDY OF PROPERTIES OF Ti BASED COATINGS DEPOSITED BY SELECTED PVD TECHNIQUES

Daniel KOTTFER[*], Milan FERDINANDY[**], Pavol HVIZDOŠ[**], Lukasz KACZMAREK[***]

Authors: ***Doc. Ing. Daniel Kottfer, PhD.,* RNDr. Milan Ferdinandy.,** Ing. Pavol Hvizdoš, PhD.,** Ing. Lukasz Kaczmarek, PhD.******
Workplace: **** Faculty of Mechanical Engineering, Technical University in Košice,***
Address: ***Mäsiarska 74, 040 01 Košice, Slovak Republic***
Workplace: ***** Institute of Materials Research, Slovac Academy of Science***
Address: ***Watsonova 47, 040 01 Košice, Slovak Republic***
Workplace: ****** Instytut Inzinierii Materialowej, Politechnika Lódzka***
Address: ***ul. B. Stefanowskiego 1/15, 90-924 Lódz, Poland***
Phone: ***+421556023510***
E-mail: ***daniel.kottfer@tuke.sk; mferdinandy@imr.saske.sk; phvizdos@imr.saske.sk; lukaszkaczmarek78@gmail.com;***

Abstract

Mechanical and tribological properties of thin TiN, TiAlN and KTRN coatings applied by two PVD techniques were investigated. PVD techniques of ARC and SARC were used for the deposition of thin coatings onto HSS Co5 steel substrates. Conventional types of coatings (TiN and TiAlN monolayers) and an advanced type of coating (Ti and Al based KTRN monolayer deposited using SARC) with smaller microdrops on the surface were analyzed by standard techniques in order to asses the surface status and quality – coating thickness, chemical composition, nano-hardness, Young´s modulus, and tribological properties at room temperature. Nanohardness values of TiN, TiAlN and KTRN coatings were 31.33GPa, 26.05GPa and 30.42 GPa, respectively. Coefficient of friction (COF) values of TiN, TiAlN and KTRN coatings were 0.86, 0.49 and 0.63, respectively. The roughness R_a of TiN, TiAlN, and KTRN coatings was 0.71µm, 0.33 µm, and 0.66 µm, respectively. High roughness and larger contact surface area of the system coating-ball are reasons of high COF of TiN coating.

Key words

PVD coating; wear; COF; nano-hardness;

Thin coatings are used to prolong the lifetime of industrial parts. Properties and the applications of TiN coatings have been studied extensively (1-13). The addition of other elements such as Al, Cr, etc., increase the oxidation resistance at the temperatures above 450 °C. TiAlN coatings wre developed for engineering applications as possible alternatives to TiN coatings (13-18).

The next logical step in improving properties (above all hardness, COF, and refractoriness) was the development of TiAl based coatings as thermal barriers (19).

The mentioned layers are usually deposited by CVD (11, 20) and by PVD techniques (1,5,12,14,16,18-21). The most promising methods are: ARC and LARC PVD (Lateral Rotating Arc-Cathodes) (2,6,12,14,16) but also activated evaporation by EB PVD (12,13,21,22).

The aim of this paper was to determine the tribological properties of the hard wear resistant thin coatings deposited by the PVD processes onto the high speed steel Co 5. Use of complementary evaluation methods of various mechanical properties evaluation can give us comprehensive information about the quality of applied coatings. The next aim of the article is to compare the results to the data given by producers of the coatings as well as to those reported by the above-mentioned authors.

Preparation of specimens and experimental procedure

Experiments were focused on the evaluation of the coating properties and were carried out using ***specimens*** of HSS Co5 (STN 19852, AISI M35) high speed steel which are used for drill bits fabrication. The HSS Co 5 containing C = 0.92 %, W = 6.40%, Mo = 5.0 %, V = 1.90 %, Co = 4.80 % and Cr = 4.10 %, was used as the substrate material. The specimens were heat-treated in the salt bath furnaces. They were austenized at the temperature of 1180 °C and triple tempered at the temperature of 540 °C. After this heat treatment the steel acquires hardness of 65 HRc (STN 19852, AISI M35). The specimens were ground before depositing the coatings to roughness R_a = 0.4 – 0.6 μm. Coated specimens for experiment were supplied by Staton company.

Differently shaped specimens were prepared according to the requirements of specific testing procedures. For tribological pin-on-disc testing, the flat planparallel discs with 30 mm diameter and 5 mm thickness were prepared. Specimens for measuring coating thickness had the dimensions of 6x5x35mm. Roughness was measured using the specimens for pin-on-disc test.

All evaluated coatings were deposited in Staton company. TiN (Fig. 1) and TiAlN (Fig. 2) coatings were ***deposited*** by ARC PVD method and KTRN (Fig. 3) was deposited using SARC PVD method according to parameters shown in Table 1. Deposition time for all coatings was 30 min., specimens rotated, rotation speed was 4 - 5 rpm, the specimen-cathode distance was 20 cm. The specimens were placed on one side horizontally so that deposition took place on one side only. Before the deposition substrates were cleaned by ultrasonication in acetone and subjected to Ar plasma etching – P = 0.2 Pa, U = 1.2 kV, t = 20 min. and heating – P = 5 Pa, U = 1.24 kV, t = 60 sec.

The thickness and chemical composition of the investigated coatings and substrate were determined by scanning electron microscope JEOL 7000F and EDX analysis. Thickness was measured on brittle fracture cross sections of the coatings. The specimens were cleaned in an organic solvent by ultrasonication during 5 min and then dried using a hot air blower for 3 min.

The surface roughness investigations of substrates and deposited coatings were made on a Surftest SJ-310 contact stylus profiler by MITUTOYO.

The microhardness tests of coatings were made using CSM NHT ultra microhardness Tester with Berkovich indenter tip. Test conditions were selected so that the values at penetration depth smaller than 0.1 of the coating thickness could be measured. Under such circumstances the influence of the substrate on the measurement results can be eliminated and hardness and elastic modulus of coating can be evaluated. The measurements were made using sinus mode of loading with maximum load 0.07 N.

Tribological tests were carried out on the HTT CSM "Pin-on-disk" tester in the following conditions: counter-specimen – a fixed ball – was made of 100Cr6 steel with the 6 mm diameter and hardness HV 850 (66 HRc in agreement with ASTM E 140-97). Normal loading was 0.5 N, radius was 4 mm, nominal sliding speed was 0.1 m/s, overall sliding distance was 50 m, ambient temperature 20 °C, and humidity was 40 %. Pin-on-disc test was performed without lubrication. Friction force was continuously measured and COF was calculated and recorded.

The wear damage was expressed as the worn surface volume loss. The wear track cross section was found using Surftest 301 contact stylus profilometer by Mitutoyo and, from the area of the wear track cross section was calculated (Figs. 7, 8). The lost volume was then calculated using equation $V=S.l$, where V is volume of the worn material (mm^3), S is area of the wear track cross section (mm^2), l is sliding circle length. The sliding circle length was calculated using equation $l=2\pi r$, (mm), where r is radius of the sliding circle (mm).

MAIN PROCESS PARAMETERS FOR THE TiN, TiAlN AND KTRN COATINGS DEPOSITION

Table 1

Coating	**Technique**	**Pressure [Pa]**	**Temperature [°C]**	**I_{catode} [A]**	**U [V]**	**Flow N_2 [cm^3min^{-1}]**
TiN	arc PVD	0.2	350	80	-150	120
TiAlN						
KTRN	Sarc PVD	0.1	400	100	-200	100

Results and discussion

Thickness and hardness

Thickness of the experimental TiN, TiAlN and KTRN (Fig. 1-4) coatings was 2.0 μm, 3.0 μm and 2.1 μm, respectively. SEM micrographs of the evaluated coatings together with their EDX analyses are shown in Figs. 1 - 3.

Hardness of the substrates made of HSS Co 5 high speed steel was 7.48 - 7.75 GPa (STN 19852, AISI M35). PVD of the coatings onto the substrates lead to the increase of hardness of the coated surfaces to the values from 26.05 GPa to 31.33 GPa (Fig. 4), which in the case of TiN and KTRN is a fourfold increase in comparison with the substrate itselv. The highest hardness 31.33 GPa was observed in the case of the TiN coating. It was found out, that it was considerably higher than the value given by the producer (STATON Co, Slovakia) 23 GPa or than that measured in (8,14) where values 25.2 ± 2.8 GPa and 22.12 GPa, respectively, were found.

Yasuo Tanno and Akira Azushima (7) found the hardness of TiN (111) coating to be 25.1 GPa, Young´s modulus 300.2 GPa, which is less than our findings. Their COF for sliding distance 10 m and counterpart Al_2O_3 ball was 0.2, which is far less than our finding.

Hardness of the TiAlN coating was 26.05 GPa, 20 % lower than gives the producer (STATON company) and 2 GPa lower than (13) where hardness of TiAlN top coat deposited onto TiN coating was 28 GPa. The measured value of the hardness of the TiN coating presented in this study (31.33 GPa) is higher that in (5) where values of hardness were from 16.3 GPa to 29.8 GPa in independence on the distance of specimens from the disc. Hagarová and Štěpánek reported hardness of TiAlN coating deposited by arc PVD to be 26.7 – 29.2 GPa (13) which agrees with our results.

Hardness of the KTRN coating was 30.42 GPa, which is 5 GPa less than the producers (Staton company) data. This lower value can be caused by deviations from the exact technological parameters, particularly the deposition temperature.

The values of the roughness of TiN, TiAlN and KTRN coatings were equal to 0.71 µm, 0.33 µm and 0.66 µm, respectively.

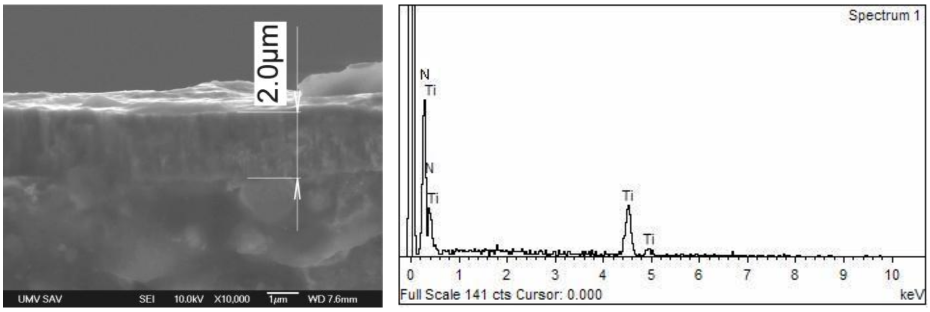

Fig. 1 *Brittle fracture (micrograph) and EDX spectrum of TiN, thickness 2.0 µm*

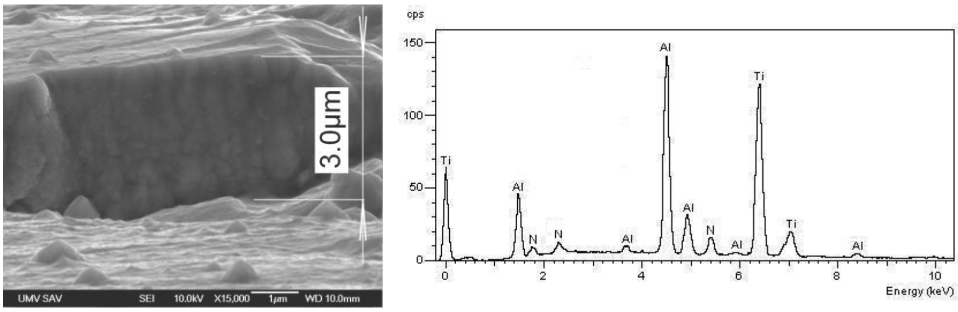

Fig. 2 *Brittle fracture (micrograph) and EDX spectrum of TiAlN coating, thickness 3.0 µm*

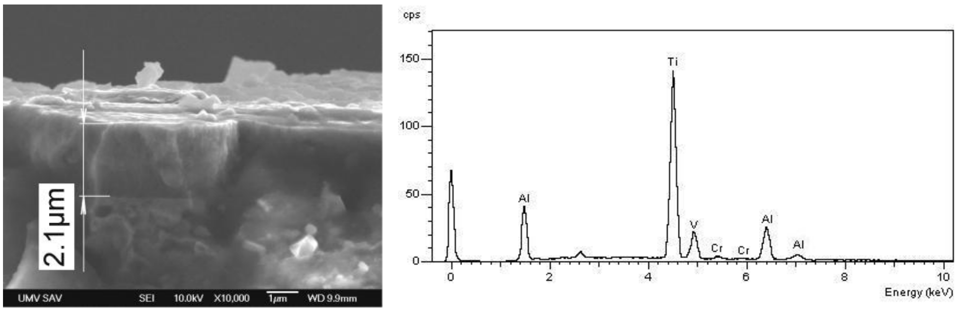

Fig. 3 *Brittle fracture (micrograph) and EDX spectrum of KTRN coating, thickness 2.1 µm*

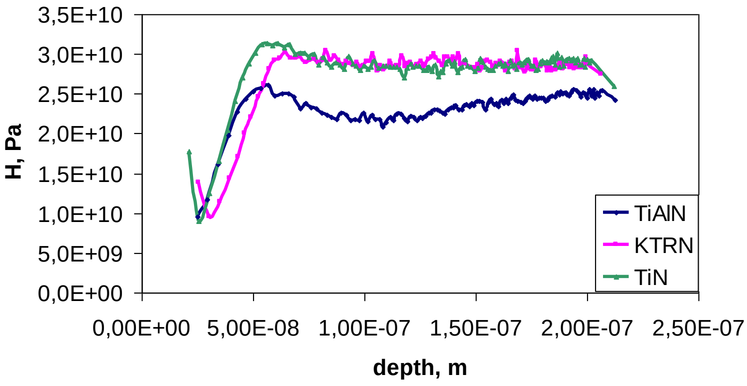

***Fig. 4** The hardness of TiN, TiAlN, and KTRN coatings*

COF and wear

As shown by COF vs. Sliding distance plots (Fig. 5), the value of COF for TiAlN (0.49) is higher in comparison with (16,17,19), where COF around 0.1, 0.2 and 0.4 were found.

B. Podgornik et al measured COF of TiAlN coating deposited on four different materials (16). Values of COF were 0.2.

COF of the tested KTRN coating (0.63) is higher than given by the producer (0.35). COF of the tested TiN coating (0.86) is also considerably higher than that given by the producer (0.4) and is comparable to results of (22), where COF around 0.8 were found. This can be due to high roughness (R_a=0.66 μm) of the coated surface.

Fig. 5 presents the COF of evaluated coatings depending up sliding distance. The COF of the KTRN coating grew markedly after traveling 5 and 10 meters and then grew to its maximum. In the case of the TiAlN coating COF kept slightly growing to its maximum. The COF of the TiN coating grew linearly after traveling 12 meters and then grew gradually to its maximum. COF was influenced mainly by the roughness Ra, coating hardness, and the contact surface area.

Wear was evaluated in terms of material volume loss during Pin-on-disc test. Values of the wear of TiN, TiAlN and KTRN coatings were equal to 0.60 x $10^{-3}mm^3$, 1.76 x $10^{-3}mm^3$ and 0.817 x $10^{-3}mm^3$, respectively. With respect to thickness of the coatings it can be seen that the depth of the wear tracks ***b*** did not exceed the thickness of the protective coatings (Fig. 6, 7). The above-mentioned figures show the width of the wear track ***a***, which is in a good agreement with volume loss of evaluated coatings. Wear – coating volume loss – was lowest for TiN coating. On the other hand, its values of COF were the highest ones. The COF growth was caused by increasing contact area of the coating – counter-specimen system. The wear of counterpart was not investigated.

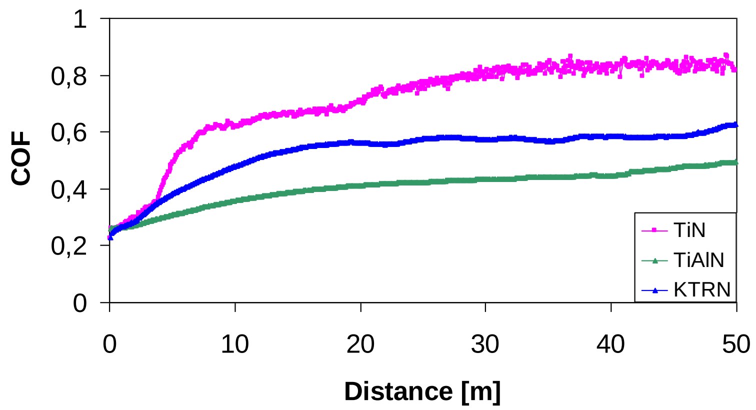

Fig. 5 *COF of TiN, TiAlN and KTRN coatings*

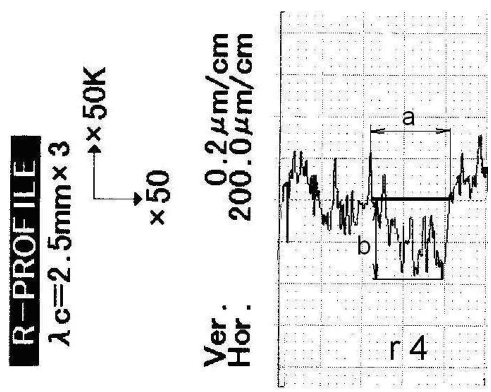

Fig. 6 *Profile of cross sections of the wear tracks of TiN coating after Pin-on-disc testing: a- width, b-depth, r4-radius of sliding track*

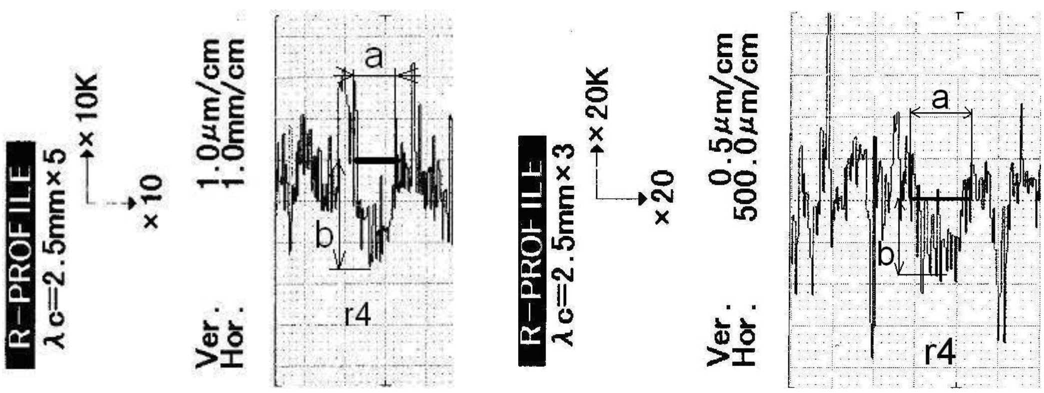

Fig. 7 *Profiles of cross sections of the wear tracks of TiAlN coating (left) and KTRN coating (right) after Pin-on-disc testing:*
a-width, b-depth, r4-radius of the sliding track

Conclusions

According to the measurements and obtained results, the following conclusions can be drawn:

- Measured values of hardness are different from the values mentioned by producer. The highest values of the hardness for TiN and KTRN coatings were 31.33 GPa and 30.42 GPa, respectively. In the case of KTRN it is about 5 GPa less than the values given by the producer. The hardness of the TiN coating was higher than that given by the producer and higher than that reported by the above-mentioned authors. The lower hardness of the KTRN coating can be caused by its irregularities (lower coating smoothness and voids within), and also by higher roughness R_a of the substrate and errors in the deposition parameters.
- COF was measured along sliding distance of 50 m. For TiN, TiAlN and KTRN coatings, the mean values of COF were 0.82; 0.48 and 0.56, respectively. The highest COF had TiN, the lowest TiAlN coating. The higher value can be a consequence of the higher coating roughness R_a. Measured values of COF differ from the values given by the producer. It can by due to different parameters of the Pin-on-disc test because the producer does not specify them.
- The wear was measured at sliding speed 10 cm/s in terms of volume loss. The TiN coating suffered the lowest wear. On other hand COF of TiN coating was high (0.86). It can be due to higher hardness of the TiN coating and lower hardness of the counter-specimen. The wear of the counter-specimen was not investigated. The highest wear was found for the TiAlN coating which had the lowest COF.

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Reviewers:

Prof. Ing. Peter Jurči, PhD.
Doc. RNDr. Pavol Šuta, PhD.