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Characterization of Al Based Coatings Deposited by PVD Technique onto Samples Made from High Speed Steel

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Abstract. The paper deals with evaluation of selected properties of TiAlN and CrAlN coatings deposited by PVD technique on specimens from the high speed steel HS6-5-2-5 (STN 19 852). There were evaluated tribological properties such friction coefficient and wear as well as mechanical properties such thickness and chemical composition of coatings, hardness and Young´s modulus. These Al based coatings were tested by selected methods. Measured properties were compared to other authors.
Keywords: materials engineering**,** Al based coatings, PVD technique, properties

1. INTRODUCTION

Thin coatings are often used for the lifetime prolongation of engineering components. Thin Ti based coatings are widely used due to the good properties like adherence, hardness, wear rate and friction coefficient as coatings of cutting tools [1, 2]. Thin Ti and Cr based coatings are widely used as monolayers [1-6], multilayers [6-10] and gradient layers [5].

Hagarová and Štěpánek studied TiN and TiAlN coatings deposited by arc PVD process with different values of accelerating bias voltage. Properties such as thickness, hardness, adhesion were evaluated [1].

Jakubéczyová et al. investigated the properties and cutting performances of two thin coatings. PVD techniques arc and larc were used for the deposition of thin AlTiCrN and nACo coatings onto cutting tools prepared by powder metallurgy [2].

Chang-Lin Liang et al. studied the techniques of synthesizing the properties of TiN and TiAlN, and moreover improving the performances of the coatings with nanometer period structures, prepared by magnetic filtered pulsed vacuum cathodic arc plasma technique (MFPVC arc PVD) [4].

Podgornik et al. evaluated three commercial PVD/PACVD hard coatings deposited on ~2 µm thick TiAlN support layer [5].

Tlili et alinvestigated the effect of aluminum content on the morphology and structure of CrAlN coatings which were deposited onto silicon (100) substrate using an unbalanced magnetron sputtering PVD technique in a reactive nitrogen atmosphere at aluminum applied negative voltages (0, –300, –500, –700 and –900 V). Deposited coatings were contamined by Al with content of Al 0, 5, 13, 28 and 30%, the thicknesses were 1.8, 2.1 2.5, 2.7 and 3.0 µm, the hardness was 24, 26, 26, 23 a 30 GPa, Young´s modulus was 400, 410, 410, 380 a 460 GPa, respectively [6].

Endrino and Derflinger evaluated the influence of alloying elements on the phase stability and mechanical properties of AlCrN coatings deposited using the reactive cathodic arc deposition technique [7].

Dobrzanskiet al. studied structure and properties of the multicomponent TiAlSiN coatings obtained in the PVD process in the nitride tool ceramics. TiN coating and TiN+coatings+TiN systems were also evaluated [8].

Transition metal nitrides were investigated widely due to their excellent intrinsic properties such as good conductivity, high hardness and wear resistance, being applied as diffusion barriers, hard or wear resistant coatings and anti-corrosion coatings [7, 10]. The properties and applications of TiN coatings have been studied extensively. The addition of other elements such as Al, Cr, etc. increases oxidation resistance over 450°C. TiAlN coatings have been developed for engineering applications as an alternative to TiN coatings [1, 4]. Showed coatings are deposited by CVD techniques [5, 6, 9] and by PVD techniques.

The aim of this work was to determine the tribological properties of the hard, wear resistant and thin coatings obtained in the PVD process on the high speed steel HS6-5-2-5 (STN 19 852). These Ti and Al based coatings were tested by selected methods. These methods are in correlation, and together they can give us information about the quality of applied coatings.

2. PREPARATION OF SPECIMENS AND EXPERIMENTAL PROCEDURE

Experiments were made using specimens from the high speed steel HS6-5-2-5 (STN 19 852) containing 0.96% C, 4.5% Cr, 5.2% Mo, 6.7% W, 2.0% V, and 5.0% Co. The steel obtains a hardness of 63-66 HRC after its heat treatment. The specimens were mechanically polished before deposition of coatings for roughness R_a = 0.4-0.6 μm.

Deposition parameters were as follows. Coatings were deposited by Sarc PVD technique, Sarc meaning arc from Staton deposited with smaller microdrops designed in [2] according to these parameters: temperature from 400 to 500°C, deposition time was 30 min., spacing current was 60-100A, pressure was 0.1-0.5 Pa. Specimens rotated during the deposition process, coating was deposited onto one surface of specimens, the rotation speed was 4-5 rpm, the distance of specimens to the cathode was ~ 20 cm. The specimens were polished forroughness was R_a = 0.35 μm. Before coating deposition substrates were cleaned ultrasonically 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 compound of the investigated coatings was determined by scanning them with the electron microscope Jeol 7000F and XRD analysis. The thickness was determined on fracture of specimens after they were cooled in liquid nitrogen. Specimens were cleaned in an organic solvent ultrasonically; they were then cleaned for 3 min and then dried by warm air. The microhardness tests of coatings were made using the CSM microhardness tester. Test conditions were selected so that the penetrate depth was to 0,1 of the thickness of the evaluated coatings. Measurements were made at 0.07N load and frequency of the load 20 Hz eliminating any influence of the substrate on the measurement results.

Tribological tests were carried out on the CSM “pin-on-disk” tester in the following conditions: counter-specimen – ball made from the 100Cr6 steel ball with the 6mm diameter, counter-specimen load – 0.5 N, friction radius − 2 mm, linear velocity – 0.05 m/s, respectively, and an ambient temperature of − 20°C. The character of the developed failure was not evaluated.
For the determination of wear depth (Fig. 8, 9) after friction test was used apparatus Mitutoyo Surftest 301. The result of Pin-on-disc test is coefficient of friction (COF), Tab. 1.

Table 1. Main process parameters for the TiN and TiAlN coatings depositon

	Technique	Thickness [μm]	Pressure [Pa]	Temperature [°C]	I_{catode} [A]	U [V]	Flow N_2 [$cm^3 min^{-1}$]
CrAlN	Sarc PVD	2.1	0.2	400-500	80	-150	120
TiAlN		1.0					

3. RESULTS AND DISCUSSION

Thickness and chemical compositions of evaluated coatings were measured by scanning them using the electron microscope Jeol 7000F. The thickness of the CrAlN coating (Fig. 1) is 1.1 μm and the TiAlN coating (Fig. 3) is 1.9 μm. The chemical composition of these coatings were determined by EDX analysis (Fig. 2, 4) shown in Tab. 2, 3.

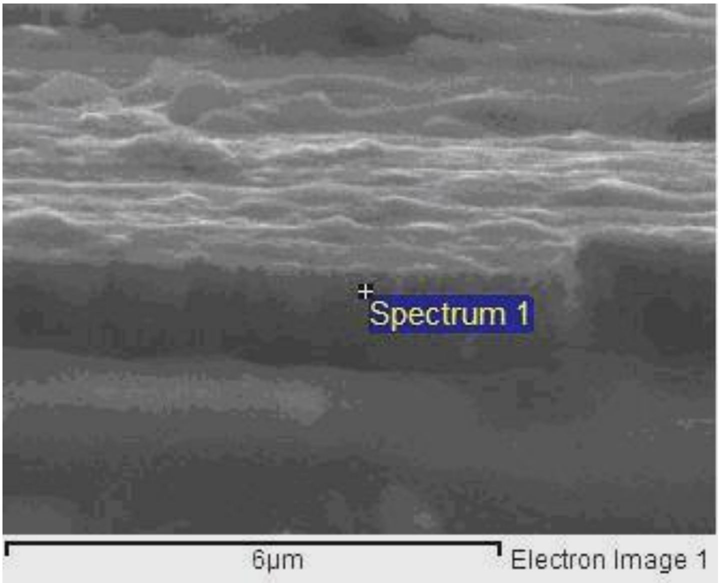

Fig. 1. Structure of CrAlN coating, SEM

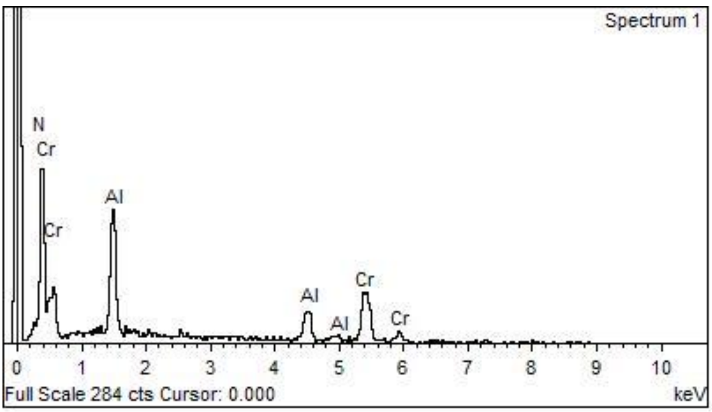

Fig. 2. Chemical composition spectrum of CrAlN coating

Table 2. Quantitative EDX analysis of CrAlN coating

Elmt	Element %	Atomic %
Cr K	49.04	28.23
Al K	21.91	15.53
N K	29.15	56.24
Total	100.00	100.00

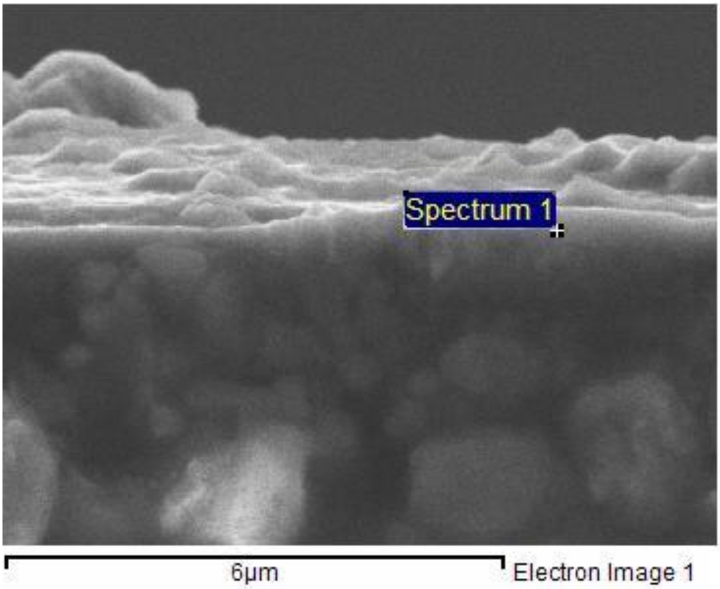

Fig. 3. Structure of TiAlN coating, SEM

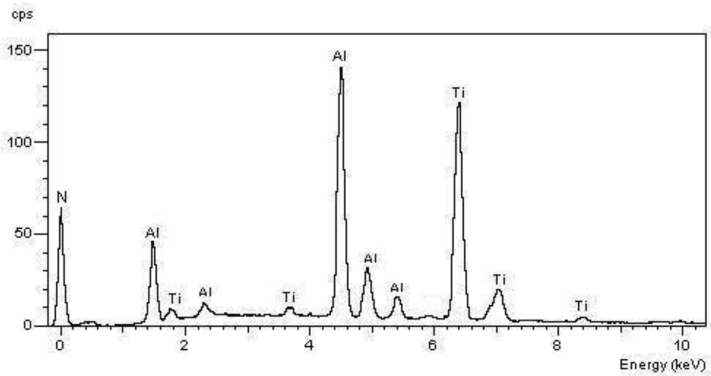

Fig. 4. Chemical composition spectrum of TiAlN coating

Table 3. Quantitative EDX analysis of TiAlN coating

Elmt	Element %	Atomic %
Ti K	21.43	23.12
Al K	52.21	56.56
N K	26.36	20.32
Total	100.00	100.00

The hardness of the evaluated coatings were made by apparatus NHT of CSM Instruments company using nanoindentation method in sinusoidal mode with frequentional loading 20 Hz, and maximum load 300 mN. Berkovich indenter was used. Young´s modulus was evaluated as a part of the hardness measurements. The hardness (Fig. 5) of CrAlN coating is 17.55 GPa and TiAlN coating is 14.63 GPa. Young´s modulus (Fig. 6) of CrAlN coating is 590.09 GPa and TiAlN coating is 586.45GPa.

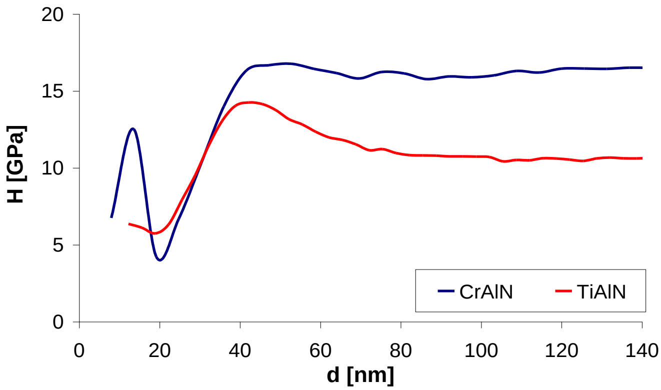

Fig. 5. Hardness of of CrAlN and TiAlN coatings

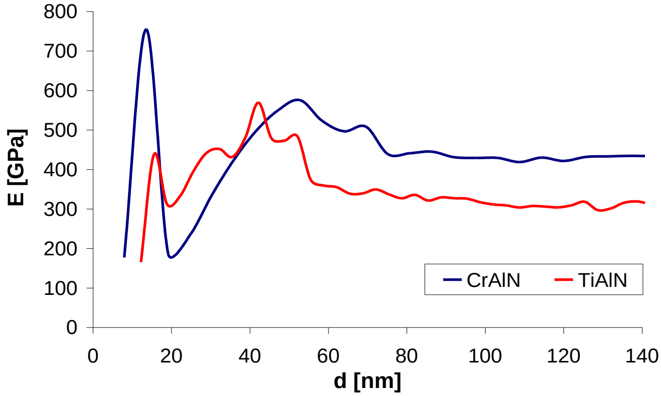

Fig. 6. Young´s modulus of CrAlN and TiAlN coatings

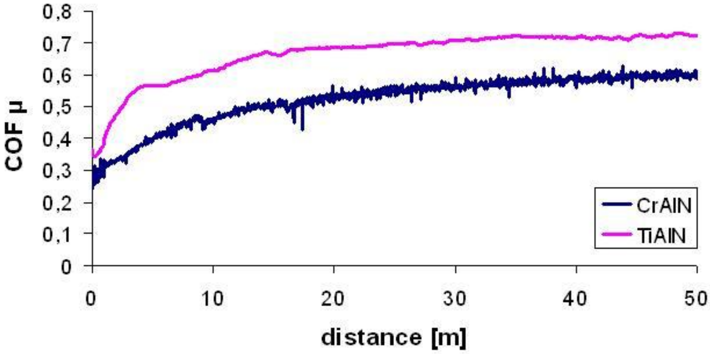

Fig. 7. COF of TiAlN and CrAlN coatings, load 0.5 N, speed 5 cm/s

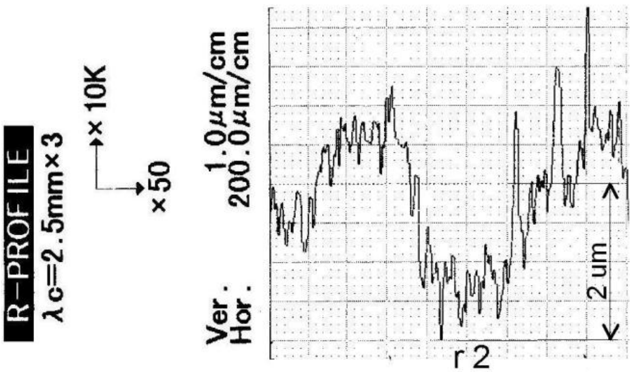

Fig. 8. Profile record of CrAlN layer from profilomer after Pin-on-disc teste, radius r = 2 mm

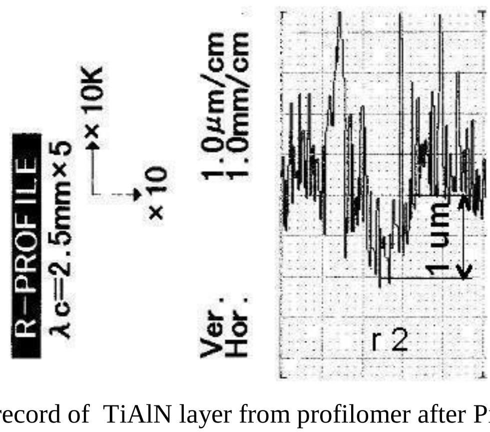

Fig. 9. Profile record of TiAlN layer from profilomer after Pin-on-disc teste, radius r = 2 mm

The wear and COF of CrAlN and TiAlN coatings were evaluated by a Pin-on-disc test. The COF of CrAlN and TiAlN coatings (Fig. 8, 9) was 0.72 and 0.58, respectively. The wear rate was evaluated as a depth of wear using a profile device. The wear profile of coating was carried out with transition of indentor of profilomer in the perpendicular direction on the ball movement. The wear depths of CrAlN and TiAlN coatings were 2.0 a 1.0 μm, respectively (Fig. 10). Based on the results, wear material was mostly transferred to border of wear trajectory. The contact surface between the coating and counterpart increases and due to increases COF. In the case of the CrAlN, COF coating increases from 0.35 to 0.55 after 5 m. From this value, COF increases to maximal value 0.72 gradually (Fig. 7). On the other hand, COF of the TiAlN coating increases from 0.25 to maximal value 0.61 (distance 50 m) gradually (Fig. 7).

The COF of TiAlN coating is lower than COF of CrAlN coating. The wear rates (depths) of CrAlN and TiAlN coatings are from 1.5 to 2.0 μm (Fig. 8) and 1.0 μm (Fig. 9), respectively.

Measured properties of evaluated coatings are showed in Table 4.

Table 4. Mechanical properties and friction coefficient of evaluated coatings

	Thickness [μm]	Hardness [GPa]	Young´s modulus [GPa]	Roughness R_a [μm]	COF v = 5 cm/s
CrAl	1.1	17.55	590.09	0.76	0.72
TiAlN	1.9	14.63	586.46	0.31	0.61

4. CONCLUSIONS

Two Al based coatings, deposited by Sarc PVD technique, were evaluated. For a consideration measured properties can be say following:

- thickness, roughness, hardness, Young´s modulus, wear rate and COF were evaluated
- thickness of evaluated CrAlN and TiAlN coatings (Fig. 1, 3) were comparable with values of CrAlN and TiAlN coatings presented in literature
- measured values of hardness of CrAlN and TiAlN coatings were 17.55 GPa a 14.63 GPa (Fig. 5), respectively. Hardness of CrAlN coating is lower than maximal values of [6, 7, 11]. Hardness of TiAlN coating is lower than maximal values measured by Hagarová et all [1]. Young´s modulus of CrAlN and TiAlN coatings were 590.09 and 586.46 GPa, respectively (Fig. 6). Showed values are fundamentally higher than measured values by Chang-Lin Liang et all. [4] and Tilli et all. [6]

- COF of CrAlN and TiAlN coatings were 0.72 and 0.61, respectively (Fig. 7). COF of CrAlN coating is higher than COF of TiAlN coating. Wear rate was evaluated as a depth of wear surface. Wear rate of CrAlN coating is higher than wear depth of TiAlN coating.

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