RESEARCH OF SELECTED PROPERTIES OF CR BASED COATINGS DEPOSITED ON HIGH SPEED STEEL BY PVD TECHNIQUE

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The paper deals with evaluation of selected properties of AlCrN and nACRo3 coatings deposited by PVD technique on specimens made of high speed steel CPM 10V produced by powder metallurgy. Tribological properties such friction coefficient (FC) and wear as well as mechanical properties such thickness and chemical composition of coatings, hardness and Young´s modulus were evaluated in the paper. These Cr based coatings were tested by Pin-on-disc test, Calotest, scanning electron microscope and EDX analysis. Maximal measured values of hardness of AlCrN and nACRo3 coatings were 31.02 GPa a 29.82 GPa, respectively. Adhesions of AlCrN and nACRo3 coatings obtained level HF2 (acceptable) and HF6 (not acceptable), respectively. Maximal values of Young´s modulus of AlCrN and nACRo3 coatings were 394.19 GPa and 308.15 GPa, respectively. FC of AlCrN and nACRo3 coatings were 0.44 and 0.84 at ball speed 5cm/s, and 0.20 and 0.54 at speed 10cm/s, respectively. Measured properties were compared with data observed by other authors and producer.

K e y w o r d s: Cr based coatings, LARC PVD technique, adhesion, friction coefficient

1 INTRODUCTION

Ternary systems, such as MeAlN (Me=Ti, Cr etc.), are attractive more and more attention from the industry. This is due to their high resistance to wear. Thin Cr based layers are often used for its properties such adherence, hardness, wear rate and friction coefficient (FC) as coatings on cutting tools [1, 2].

Transition metal nitrides were widely investigated due to their excellent intrinsic properties such as good conductivity, high hardness and wear resistance and they have been applied as diffusion barriers, hard or wear resistant coatings and anti-corrosion coatings [6].

Titanium-based coatings, especially TiAlN are industrial state-of-the-art tool coatings for most machine operations. Indeed, it is known that Al permits higher working temperatures because of the alumina thermal barrier protects the tool core during high speed machining. The addition of Al, Cr increases oxidation resistance over 450 °C. TiAlN coatings have been developed for engineering applications as an alternative to TiN coatings [1]. Therein before coatings are applied by PVD technique [2-8]. The most widespread are methods: arc PVD techniques [1-3] and magnetron sputtering PVD techniques [4-8] with using N_2 as reactive gas in the process of deposition. The development of TiAlN or CrAlN hard coatings can be carried out by magnetron sputtering PVD technique via varying the aluminum target bias voltage to improve the properties of the traditional TiN and CrN [8].

Jakubéczyová et. all investigated properties and cutting performance of thin AlTiCrN and nACo coatings onto cutting tools prepared by powder metallurgy by PVD technique [2].

OC Oerlikon Balzers produces AlCrN coatings named BALINIT® ALNOVA which are just a few thousandths of a millimetre thick but harder than steel; these low-friction coatings are extremely wear-resistant and chemically inert. Producer declared microhardness HV 0.05 of this coating 3.20 and friction coefficient (FC) against steel 0.3. For coating BALINIT® HELICA (AlCrN based coating) declares microhardness HV 0.05 and FC 3.000 and 0.25; respectivelly [3].

Souza et all researched properties of the AlCrN coating deposited onto Si_3N_4 ceramic cutting tool sintered with CeO_2 and Al_2O_3 using . They measured hardness value equal to 31 GPa [4].

Fox-Rabinovich et all studied the effect of mechanical properties measured on the wear resistance of cutting tools with TiAlN and AlCrN coatings at room and elevated temperatures. They measured hardness, Young´s modulus and adhesion values equal to 28.8 GPa; 360 GPa and about 8 N, respectively [5].

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

Endrino and Derflinger discussed the influence of alloying elements on the phase stability and mechanical properties of AlCrN coatings prepared by cathodic arc evaporation method and the targets contained 70 at.% of aluminum, 25 at.% of chromium and 5 at.% of Ti, V, Y, Nb, Mo, and W. The phase stability, mechanical properties and oxidation behaviour of the deposited coatings are discussed in conjunction with their potential in machine applications. They exhibited hardness values from 27 GPa to 35 GPa, Young´s modulus from 377 GPa to 535 GPa and adhesion HF1 to HF2 [7].

Nouveau et all compared the properties of CrAlN coatings obtained by magnetron sputtering with one (CrAl) or two targets (Cr and Al). The influence of parameters such as the target bias voltage, the working pressure, the deposition time and the bias voltage applied on the Cr or Al targets on the properties of the layers was studied. Measured values of hardness were from 15 GPa to 36 GPa; Young´s modulus were in the range (331-520) GPa and FC 0.6 -0.7. [8].

Podgornik et all investigated possibilities of the application of hard coatings for blanking and piercing tools. They investigated adhesion and tribological properties of three coatings: AlCrN, TiCN and

TiAlN+DLC. Measuremed AlCrN coating data of adhesion were equal to HF1 až HF3 and HF6; FC were 0.4-0.5 [9].

The aim of this work was to determine the tribological properties of the hard, wear resistant thin coatings obtained in the PVD process on the steel CPM-10V. AlCrN and nACRo3 coatings were tested by selected methods. These methods are in correlation, and together they can give us information about the quality of applied coatings. The next aim was to compare measured values of coating properties with producer´s data and the above-mentioned authors.

2 PREPARATION OF SPECIMENS AND EXPERIMENTAL PROCEDURES

Substrate was made of steel CPM-10V by producer Crucible in USA using powder metallurgy. CPM-10V material was the first in the family of high vanadium tool steels made by Crucible and it optimized the vanadium content to provide superior wear resistance while maintaining toughness. CPM-10V is exceptional for wear resistance and good toughness, makes it an excellent candidate to replace carbide and other highly wear resistance materials in colds work tooling applications, particular where tool toughness is a problem or where cost effectiveness can be demonstrated. The CPM process produces very homogeneous, high quality steel characterized by superior dimensional stability, grindability, and toughness compared to steels produced by conventional processes [10].
Chemical content of steel is: C=2.45%; Cr=5.25%; V=9.75%; MO=1.30; Mn=0.50%; and Si=0.90%.. mechanical properties are in the Table 1.

Table 1: Mechanical properties of steel CPM 10V

Hardness HRC	62±1
Young modulus E [GPa]	221
Impact strength [J]	30±3
Bend fracture strength [MPa]	4377

Coatings were deposited by LARC PVD technique (LAteral Rotating ARC-Cathodes, PLATIT company) described in [1, 10] and by producer (LISS Rožnov pod Radhoštem, CZ). Evaluated nACRo3 coating is Triple coating and it is composed from following layers: nACRo3 = Ti-CrN + AlTiN -+nACRO, where nACRO = (nc-AlCrN)/(a-Si3N4) [10].
The thickness of coatings was designated using Calotest method based on the standard EN 1071-2:

Thickness of coating *s* can be calculated as follows:

$$s = T - t \tag{1}$$

where *T* is the total depth of the penetration:

$$T = R - \left(\frac{1}{2} \sqrt{4R^2 - D^2} \right) \tag{2}$$

t is the depth of penetration in the substrate and it equals:

$$t = R - \left(\frac{1}{2} \sqrt{4R^2 - d^2} \right) \tag{3}$$

Then the equation (1) becomes:

$$s = \frac{1}{2}\left(\sqrt{4R^2 - D^2}\right) - \left(\sqrt{4R^2 - d^2}\right) \tag{4}$$

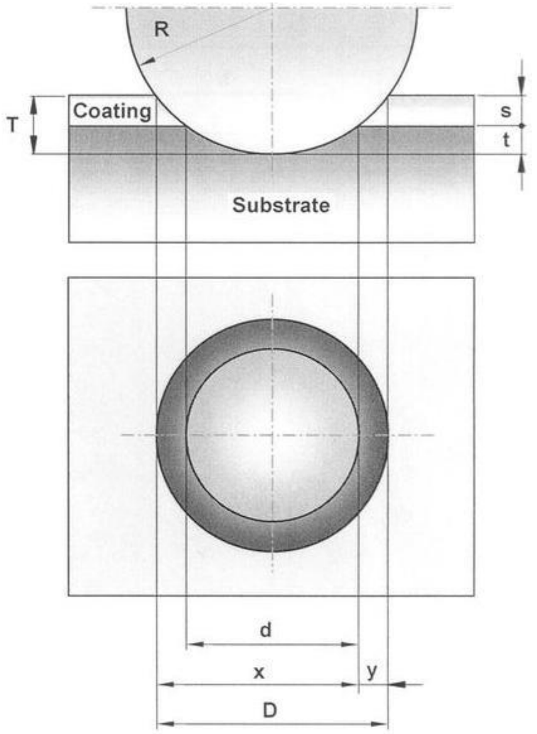

Figure 1 Dimensions of cut-out Calot (based on the standard EN 1071-2).

The chemical compositions of investigated coatings were determined by scanning electron microscope Tesla SM 340 and XRD analysis. The thickness was determined by Calotest technique and metallographically by optical microscope Olympus. The coating adhesion of the investigated specimens was carried out using the Mercedes test. The test was made by moving the diamond penetrator to the examined specimen's surface with the gradually increasing load 1.5 kN. The adhesion was made by optical microscope Olympus while border of the penetration vas evaluated and subsequently it was compared with scale which classifies the coating adhesion as HF 1 to HF 6 according to the amount of cracking or thin film coating delamination around the indent (Fig. 2).

The microhardness tests of coatings were made on the CSM nanohardness tester. Test conditions were selected so that the penetrate depth was to the limit to 0.1 of the evaluated coatings thickness. Measurements were made at 0.07 N load and frequency of the load 20 Hz eliminating 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 of the 100Cr6 steel with the 6 mm diameter, counter-specimen load – 0.5 N, friction radius −2 mm and 4 mm, linear velocity – 0.05 m/s and 0.10 m/s, ambient temperature − 20 °C, humidity – 40%. The Pin-on-disk test was carried out without lubricant. The wear rates of AlCrN and nACRo3 coatings were not evaluated.

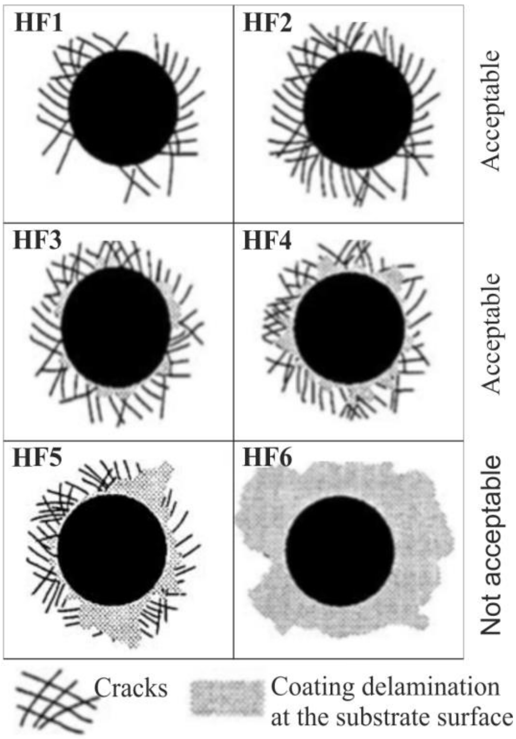

Figure 2 Scale classifying the adhesion by means of Mercedes test [10]

3 RESULTS AND DISCUSION

3.1 Thickness and chemical composition

Calotest method and optical microscope were used for evaluation of coating thickness. After measuring of calotte dimensions (Fig. 3, 6) and calculating by the help of equations (1 - 4) the thickness of evaluated coatings were determined. The chemical compositions of coatings were carried out by SEM Tesla 340SM. The thicknesses of TiAlN and nACRo3 coatings were 1.7 µm and 3.2 µm, respectively. View on the surface of AlCrN coating and EDS analysis of the point 1 are visible in the Fig. 3 and 4. Chemical composition of AlCrN coating shows Table 3.

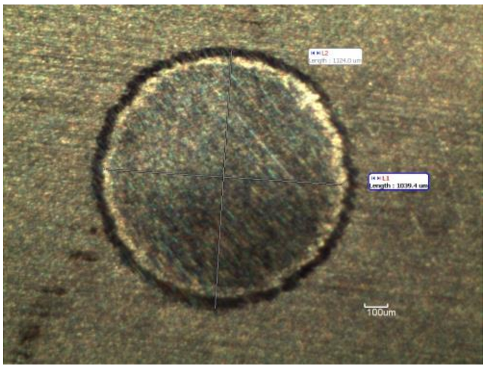

Figure 3 Polished calotte on AlCrN coating after calotest, thickness 1.7 µm.

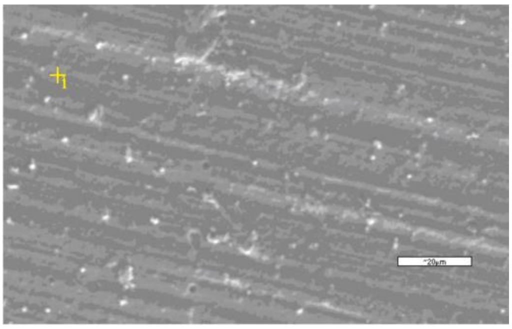

Figure 4 View on the surface of AlCrN coating.

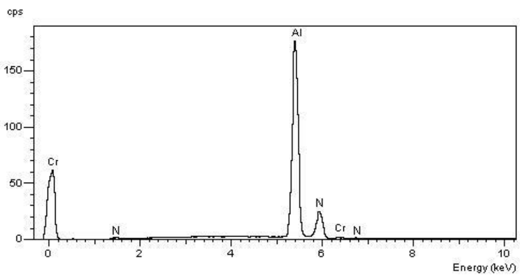

Figure 5 EDX analysis of AlCrN in the point 1.

Table 2 The chemical composition of the AlCrN coating

Chemical element	wt. %	at. %
Al (K)	43.19	48.83
Cr (K)	27.15	27.82
N (K)	29.66	23.35
Total	100.00	100.00

Polished calotte on nACRo3 coating after calottes is visible on the Fig. 6. The chemical compound of nACRo3 was carried out from surface (Fig. 8) as spectrum in point 1 (Fig. 7). Chemical composition of AlCrN coating shows Table 2.

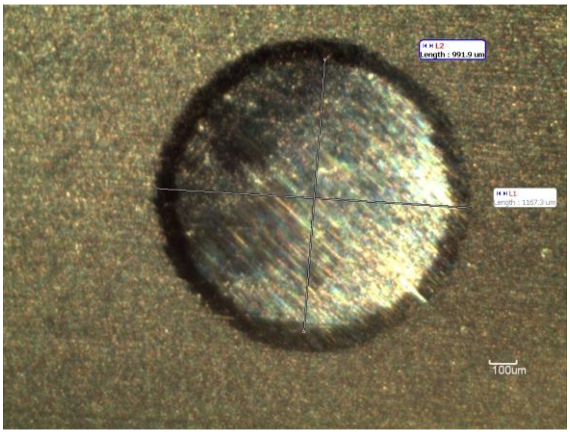

Figure 6 Polished calotte on nACRo3 coating after Calotest, thickness 3.2 µm

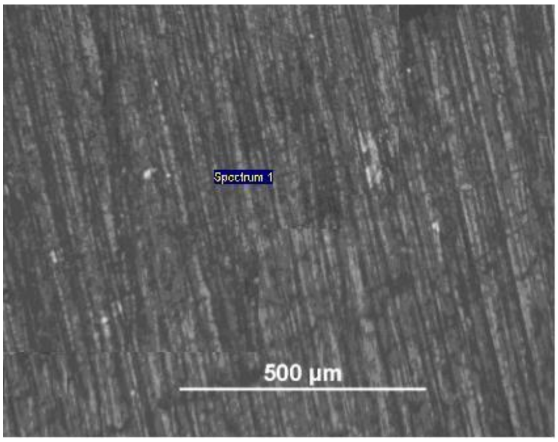

Figure 7 View on the surface of coating nACRo3.

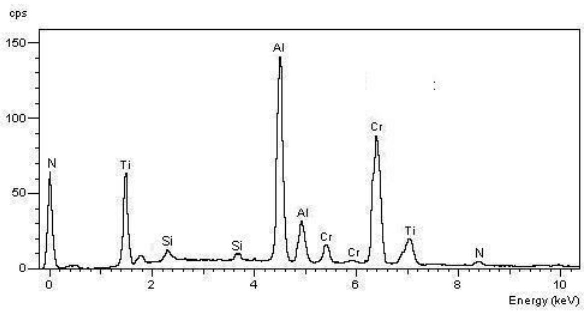

Figure 8 EDX analysis of nACRo3 layer

Table 3 Chemical composition of the nACRo3 coating

Chemical element	wt. %	at. %
Al K	37.79	35.10
Cr K	23.94	27.91
N K	21.50	22.35
Ti K	7.97	7.74
Si K	8.80	6.90
Total	100.00	100.00

3.2 Adhesion

On the basis of adhesion scale (Fig. 2), AlCrN coating was evaluated as HF6, i.e. not acceptable which corresponds to the adhesion load smaller than 20 N. Layer in the vicinity of substrate is detached which is visible in the Fig. 9. Measured adhesion stage is in good agreement with [5, 9] for the substrate as WC powder- metal. For steel substrate measured adhesion stage is far worse than in the paper [9].

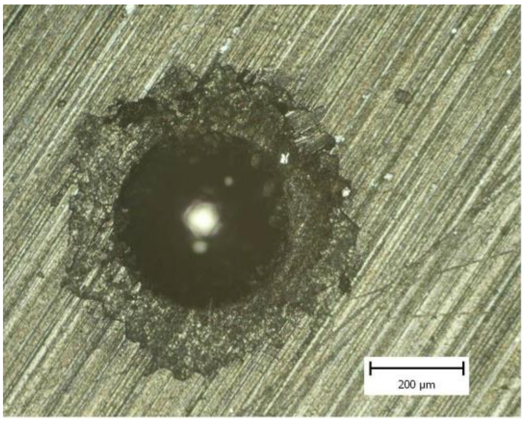

Figure 9 Mercedes test AlCrN coating on steel CPM 10V

Adhesion of nACRo3 coating was evaluated as level HF2, i.e. acceptable which correspond to the adhesion load less than 20 N. In the vicinity of indentation cracks are periodically spaced cracks in the shape of concentrated circles; coating is present in the all indentation surroundings (Fig. 10).

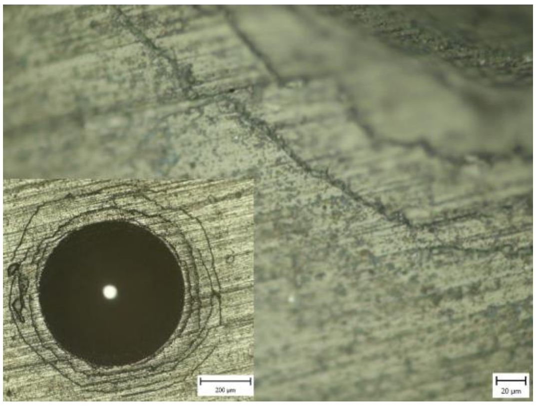

Figure 10 Mercedes test of nACRo3 coating on steel CPM 10V

3.3 Hardness and Young´s modulus

Measurement of hardness was made using nano-indentation method at sinusoidal mode with Berkowich indentor. Frequency of loading was 20 Hz and maximal load 300 mN. Young´s modulus was evaluated as well. The maximum measured values (without first part of the curves) of Young´s modulus and hardness for AlCrN and nACRo3 coatings are in the Table 4.

Table 4 The maximum measured values of Young´s modulus and hardness for evaluated coatings

	AlCrN	nACRo3
Young´s modulus [GPa]	394.19	308.15
Hardness [GPa]	31.02	29.82

Hardness of AlCrN coating is comparable with [4, 6, 8] and extensively higher than [5]; Young´s modulus was comparable with [6, 8] and higher than [5]. Value of hardness (40 and 34 GPa) for nACRo3 coating measured by producer LISS Company in CZ was rapidly lower in comparison with our value of hardness.

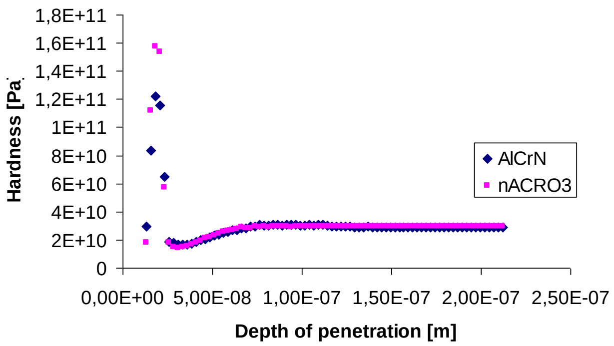

Figure 11 Hardness of CrAlN and nACRo3 coating

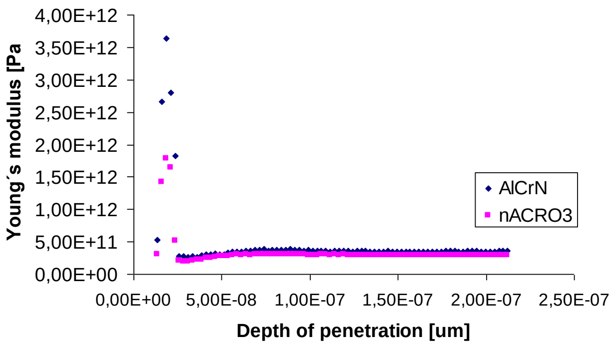

Figure 12 Young´s modulus of AlCrN and nACRo3 coatings

3.4 Friction coefficient

The FC of the AlCrN and nACRo3 coatings were evaluated by Pin-on-disc test. FC of the AlCrN and nACRo3 coatings at speed 5 cm/s were 0.44 and 0.84 (Fig. 13); at speed v=10 cm/s were 0.20 and 0.54, respectively (Fig. 14). Wear rate was not evaluated. In the case of the AlCrN coating (v=5cm/s) the FC increases from 0.10 to 0.15 after sliding distance 13 m, from this value increases FC to value 0.18 after 32m and then it increased to the maximal value 0.44 (Fig. 13). On the other hand, the FC of the nACRo3 coating was rised from 0.28 to maximal value 0.84 (distance 50m) (Fig. 13).
At ball speed v=10cm/s the FC of the AlCrN uprised from 0.10 to 0.20 continuously (Fig. 14) while the FC for nACRo3 coating increased from 0.30 to maximal value 0.54 gradually up to the distance 30 m; then FC was approximately constant (Fig. 13).

By comparison with values in [5, 8, 9] the FC for AlCrN coating is lower at speed 10 cm/s and higher than [9] at speed 5 cm/s, where coatings were deposited by magnetron sputering technique. Producer LISS Company in CZ specified FC for nACRo3 0.35.

Measured values of properties of evaluated coatings are showed in table 6.

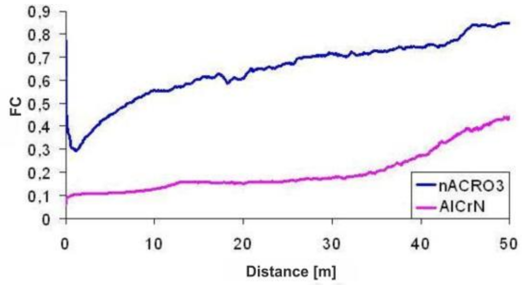

Figure 13 FC for AlCrN and nACRo3 coatings; F=0,5N; ball speed v=5cm/s

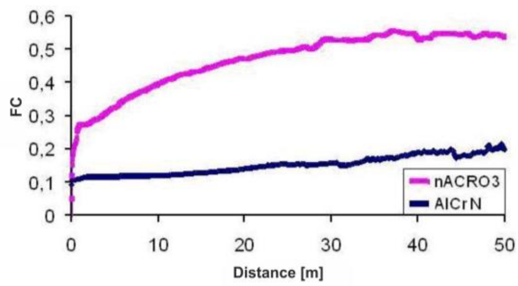

Figure 14 FC for AlCrN and nACRo3 coatings; F=0,5N; ball speed v=10cm/s

4 CONCLUSIONS

Two Cr-based coatings, deposited by LARC PVD technique, were evaluated. As for as measured properties as thickness, roughness, adhesion, hardness, Young´s modulus and FC we can say following:

- Thickness of evaluated AlCrN and nACRo3 coatings were comparable with values of AlCrN and nACRo3 coatings presented in literature by producer LISS Company.
- Roughnesses R_a for AlCrN and nACRo3 coatings achieved values 0.35 μm a 0.61 μm, respectively.
- Adhesions of AlCrN and nACRo3 coatings obtained level HF2 (acceptable) and HF6 (not acceptable), respectively.
- Maximal measured values of hardness of AlCrN and nACRo3 coatings were 31.02 GPa a 29.82 GPa, respectively. Hardness of nACRo3 coating is lower than values 40/34GPa measured by producer.
- Maximal values of Young´s modulus of AlCrN and nACRo3 coatings were 394.19 GPa and 308.15 GPa, respectively.

- FC of AlCrN and nACRo3 coatings were 0.44 and 0.84 at ball speed 5cm/s, and 0.20 and 0.54 at speed 10cm/s, respectively. FC of AlCrN coating is higher than FC of nACRo3 coating in both cases which can be caused by higher R_a. The FC of the AlCrN was lower at ball speed 10 cm/s than in [5,8,9] and higher at speed 5 cm/s as it is presented in [9].

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