THE EFFECT OF THE SUBSTRATE POSITION ON MECHANICAL AND TRIBOLOGICAL BEHAVIOR OF Ti COATINGS DEPOSITED BY EB PVD TECHNIQUE

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Abstract

Substrates with Ti coatings were deposited using the activated evaporation - electron beam physical vapor deposition (EB PVD) method. Propeties of Ti coating on substrate were investigated - mechanical properties, thickness, chemical compounds, hardness, adhesion, wear resistance. For evaluation of the surface layer mechanical properties nanoindentation and scratch test methods were used. The results are compared with bibliographical ones.

Key words

EB PVD, Ti coating, mechanical properties, wear, nanoindentation methods, scratch test

Introduction

The electron beam physical vapor deposition (EB PVD) technologies are used for aviation components` renovation, where are made from steels and Al, Mg and Ti based alloys. Coating flat surfaces with the impact of evaporated particles perpendicular to the substrate is well known [1,2]. On the other hand, depositing thin PVD layers on the inner surface of a ball and barrel is not simple. Therefore, research on the influence of technological parameters on the properties of evaporated PVD layer was realized.

There exist many materials and techniques for coating and depositing thin layers on the inner surface of the cylinder. Included among these techniques are for instance flame spraying, high velocity oxy fuel (HVOF), plasma enhanced chemical vapor deposition (PECVD), explosion deposit and electroplating. Also different materials are deposit - nitrides, borides, carbides Ti, W, V, Cr, Si for the PE CVD method or MeCrAlY (where Me = Ni, Co, Fe) for methods of flame spraying and HVOF.

Dobrzanski et al. [3] researched the structure and mechanical properties such as thickness, hardness, roughness and adhesion of PVD and CVD coatings on the basis of Ti such as Ti(C,N) deposition by the method PVD. They also studied Ti(C,N)-TiN coatings deposited by PVD method on sintered cutting devices. Then they researched the wear of cutting devices with and without Ti(C,N)-TiN coating. Bujak et al. [4] studied the mechanical properties such as thickness, hardness, roughness and the structure of PVD TiN coatings deposited by two hybrid methods: pulsed laser deposition (PLD) and magnetron-sputter-assisted pulsed laser deposition (MSPLD). The material of the substrate was HSS steel. Pawlak and Wendler [5] evaluated the TiN layer created by the hybrid PVD method including three coating deposition methods: Reactive Magnetron Sputtering (RMS), Filtered Cathodic Arc Evaporation (FCAE) and Pulsed Cathodic Arc Deposition (PCAD). Dudek et al. [6] deposited nc-TiN/a-SiN systems by plasma assisted reactive pulsed magnetron sputtering (PARPMS) from titanium and silicon targets.

As seen, Ti is an element which is used as:

- an interlayer between e.g. WC layer and steel substrate for lowering residual stresses in the system layer-substrate, the value of the Ti thermal expansion coefficient is between the values of WC and steel,
- a component of hard, wear resistant and refractory layers created by PVD methods and hybrid PVD and CVD methods.

Mechanical properties of Ti and Ti based coatings are evaluated and compared with published data. It is important to know in detail the Ti properties for the purpose of using them. Interesting are the properties of the Ti layers deposited at low substrate surface temperatures and when the incidence angle of evaporated particles is less than 90°.

The experiment was focused on the research of the influence of the substrate position on mechanical behaviour of the Ti coating deposited by EB PVD technique at temperatures under 200°C.

Experimental material and procedures

For the experiment the substrate material – steel OCHN3 MFA was selected. The material is used for production of gun barrels of the Russian production type 2A 42, 30mm calibre. Chemical composition of the electroslag remelted steel according the producer (ZTS Dubnica nad Váhom, a.s. – licensed product) is in Tab. 1. Dimensions of the samples were 6x5x30mm (Fig. 4).

Substrates were prepared before the deposition process by ultrasonic cleaning in pure acetone. Ti coating was deposited by EB PVD technique using ZIP-12 equipment (Fig. 2). Ti of the purity 99,99999% was evaporated from a sintering carbon crucible (Fig. 1). The position of the samples in the vacuum chamber was according to Fig. 3.

Parameters of the deposited coatings:

- Distance of the cathode from the Ti crucible V = 335mm
- Radial distance r =100, 110, 120, 130 and 140mm (Fig. 3)
- Pressure in the vacuum chamber 1.10^{-3} Pa
- Power of electron gun 2,5 kW
- Coating depositing time 30 min.
- Deposit temperature 200 °C.

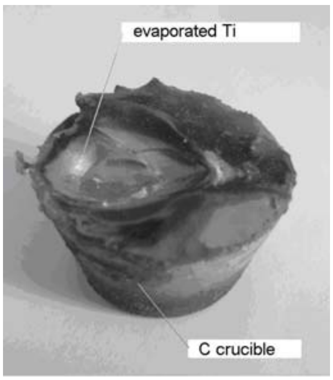

Fig. 1 *Carbon crucible with evaporated Ti*

CHEMICAL COMPOSITION OF OCHN3 MFA STEEL SUBSTRATES [wt.%] Table 1

C	Mn	Si	Cr	Ni	Mo	V	Cu	P	S
0,330	0,550	0,300	0,830	3,020	0,270	0,150	0,140	0,016	0,011

The structure and thickness of Ti coatings were studied on a brittle fracture using SEM - Jeol 7000F. The chemical composition of the coating and the substrate was evaluated by the EDX method. The sample was cooled in LN_2 and then broken so, that the Ti layer was tension stressed.

Hardness and Young´s modulus were measured by the nanoindentation tester NHT CSM Instruments with the Berkovich indenter (Fig. 7). A sinusoid mode was used with amplitude 1 mN and frequency 20 Hz, maximum load 10 mN.

Adhesion was measured by the scratch test using scratch testers CSM instruments. Measured conditions were: normal force from 0 to 120N, maximum track of the indenter 120mm.

Wear resistance of the Ti coating was measured using Pin-on-disc test.

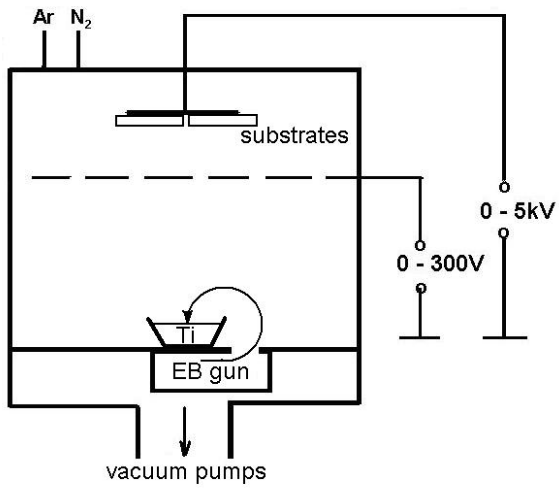

Fig. 2 *The schema of ZIP-12 equipment*

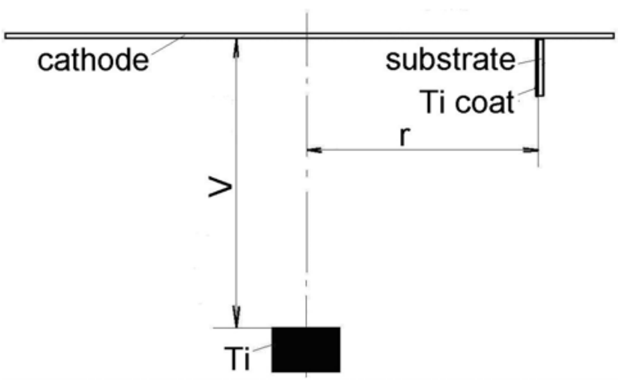

Fig. 3 *Position of samples in the vacuum chamber*

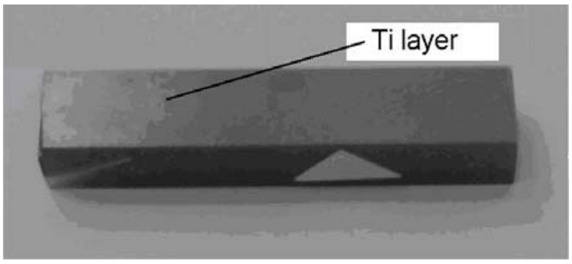

Fig. 4 *Steel sample (dimensions 6x5x30mm) with Ti layer*

Results and discussion

Thickness of the Ti layers were from 5,9 to 7,93 μm in dependence on radial distance (radius) (see Fig. 5). Ti layer with a thickness of 7,93 μm is documented in Fig. 6. For comparison, the published measured thickness if Ti layers were 2,8μm [3], 0,9 μm [4], 1,5 μm [10], the layers were deposited by PVD and hybrid methods on different substrate materials [3,4,10]. Dobrzanski et al. [3] studied the properties of Ti(C, N) + TiN layer with thickness 2,8 μm. Bujak et al. [4] measured the properties of TiN coatings with thickness of 0,9 μm deposited by CAE method and thickness of 1,3 μm deposited by CAE-PLD method. The surface of the samples with TiN coating had roughness $R_a = 0,27$ μm (CAE) and 0,1 μm (CAE-PLD). Pawlak and Wendler [5] measured the TiN layer created by hybrid PVD technology: (FCAE, RMS and PCAD) [8, 9]. Chu et al. investigated Ti/TiB2 nano-multilayers deposited onto unheated Si (100) wafers by reactive closed-field unbalanced magnetron sputtering [10]. The total film thicknesses were about 1,5 μm.

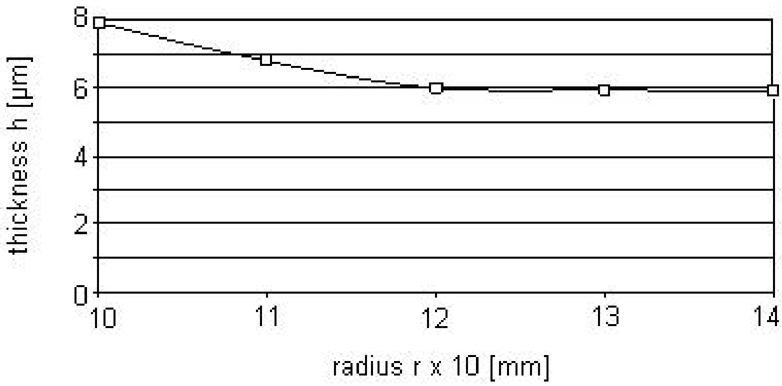

Fig. 5 *The dependence of thickness of Ti coating on the radial distance – radius of the sample in the vacuum chamber*

The coating traces the surface of the basic material (Fig. 6). In Fig. 7 is EDX analysis of the Ti layer, in Fig. 8 is EDX analysis of the steel substrate from the places displayed in Fig. 6. The coating is formed by long columns which do not grow perpendicular to the substrate. They are tilted to double the evaporated Ti particles angle to substrate (Fig. 3, 6). The dependence of Ti coating thickness on the position of the sample in the vacuum chamber is in Fig. 5.

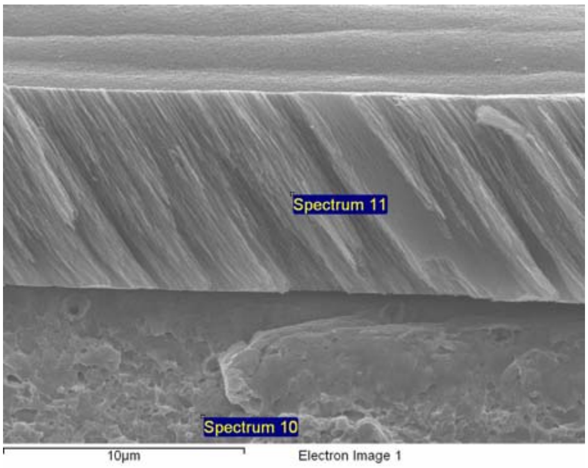

Fig. 6 *The fracture of Ti coating*

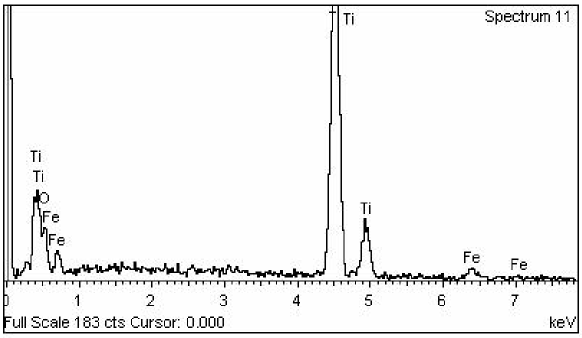

Fig. *7 EDX analysis of Ti coating*

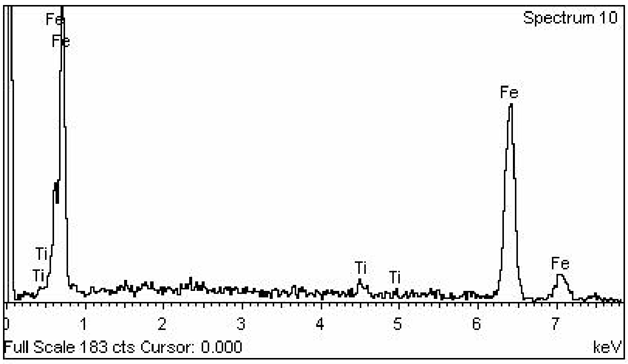

Fig. 8 *EDX analysis of steel substrate*

Nanoindetation measured hardness of Ti layers (see Fig. 9) in dependence on radial distance (radius) of substrate were from 0,65 GPa to 2,65 GPa (see Fig. 10), Young´s modulus was from 8 GPa to 53 GPa in dependence on radial distance (radius) substrate (see Fig. 11). The measured values indicate the dependence of Ti layer properties which are caused by its structure on the position of the substrate in the vacuum chamber (Fig. 10, 11).

For comparison, the published measured hardness values were 2746 HV0,05 [3], 20GPa Hardness and 308 GPa Young´s modulus of TiN layer deposited by CAE method and hardness 18GPa and Young´s Modulus 317GPa of TiN layer deposited by CAE-PLD method [4]. Pawlak and Wendler [5] studied the TiN layer created by the hybrid PVD technology: (FCAE, RMS and PCAD). Hardness of 35,4 GPa was measured, which is almost two times more than value published in [3] and hardness 16GPa of Ti layer deposited by EBPVD [8 ,9].

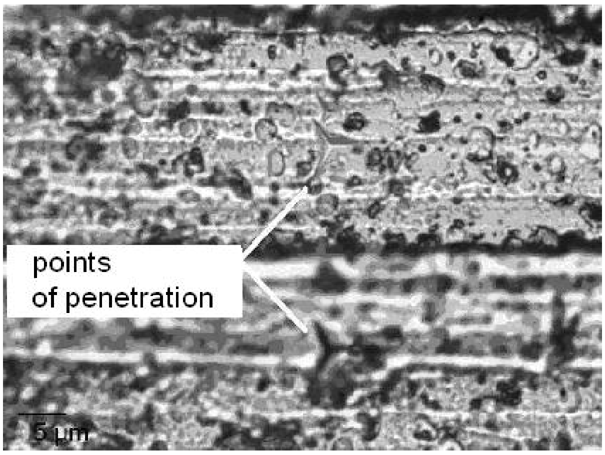

***Fig. 9** Points of penetration by Berkovich indenter to Ti coating*

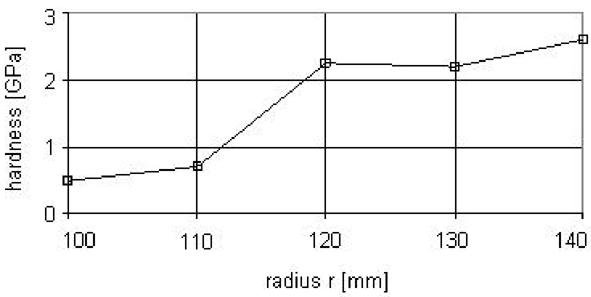

***Fig. 10** The dependence of hardness of Ti coating on the position of the sample in the vacuum chamber*

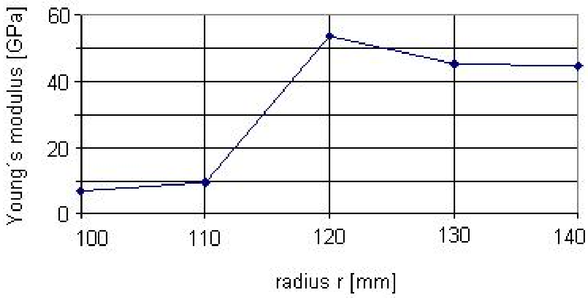

***Fig. 11** The dependence of Young´s modulus of Ti coating on the position of the sample in the vacuum chamber*

The adhesion of the Ti coating was measured by the Scratch test (see Fig. 12 and 13 – example for specimen with radial distance 100 mm). Measurements were carried out in the direction of the angle of the growth of the columns. The results were from 13 N to 33 N in dependence on radial distance (radius) of substrate in the vacuum chamber (see Fig. 14). The roughness R_a of the surface of the substrate before coating was 0,42 µm. Roughness R_a on the surface of the Ti coating was from 0,42 µm to 0,45 µm. It indicates that the Ti coating copied the microgeometry of the substrate surface.

For comparison data from Bujak et al. [4] of measured adhesion were 67 N for TiN coating deposited by CAE method and 70 N for TiN coating deposited by the CAE-PLD method. The surface of the samples with TiN coating had roughness R_a = 0,27 (CAE) and 0,1 µm (CAE-PLD).

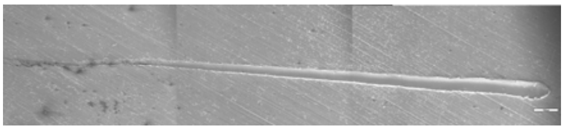

Fig. 12 *Track from the indenter after Scratch test*

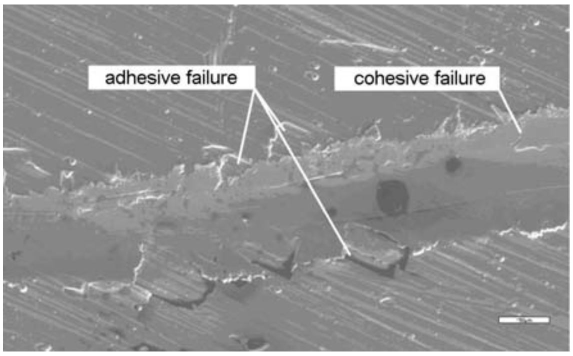

Fig. 13 *Adhesive and cohesive failure of the Ti coating*

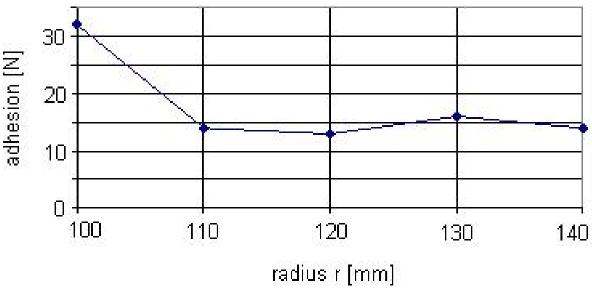

Fig. 14 *Dependence of the adhesion of the Ti coating on the position of the samples in the vacuum chamber*

Wear resistance of the Ti coating was measured using Pin-on-disc test on specimen with radial distance 140 mm. Test is illustrated in Fig. 16 and 17. Wear of the Ti coating was $7{,}564.10^{-3}$g and the counterpart (steel ball) $8{,}433.10^{-3}$g (Fig. 16). The friction coefficient was μ = 0.4 (track 50 m) and maximum value μ = 0.9 (track above 55 m to 90 m) – see Fig. 15. For comparison aaccording to Bhushan and Gupta [2] there is μ = 0.80 for the sputtered Ti layer.

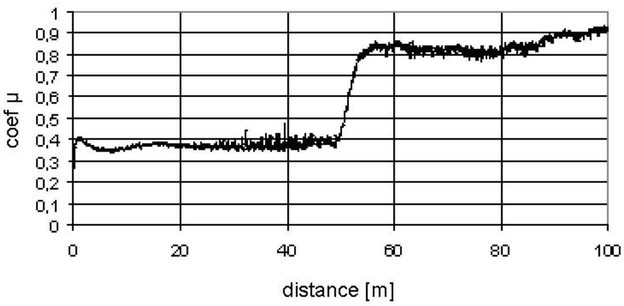

Fig. 15 *The friction coefficient Ti coating at Pin-on-disc test*

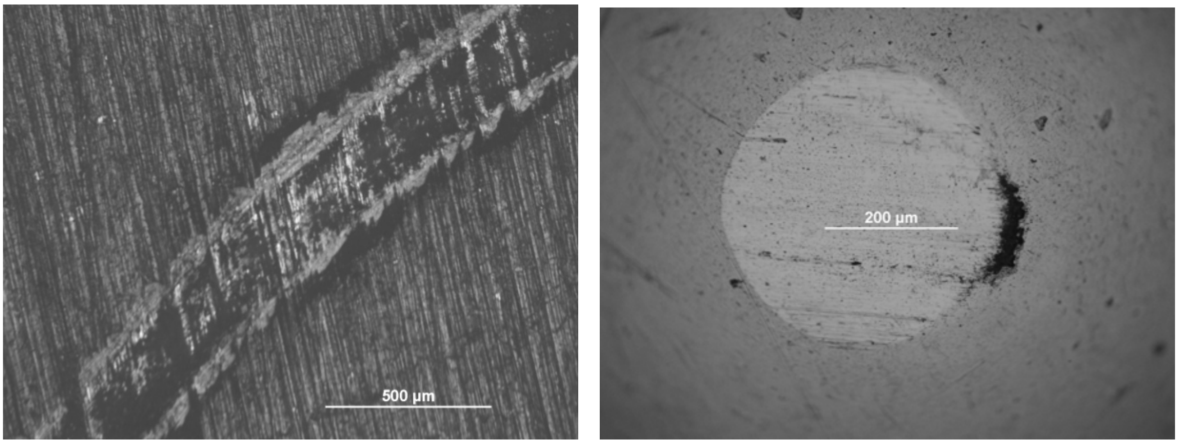

Fig. 16 *Wear of the Ti coating and steel ball after Pin-on-disc test*

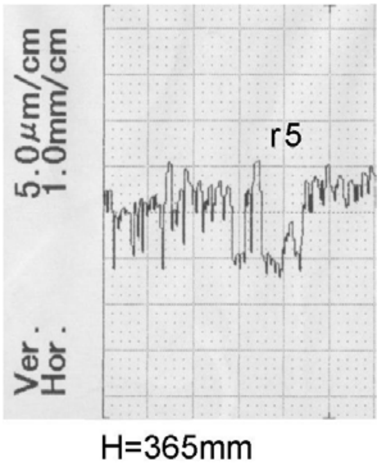

Fig. 17 *The part of the profilomer roughness, cross section of the wear on Ti coating*

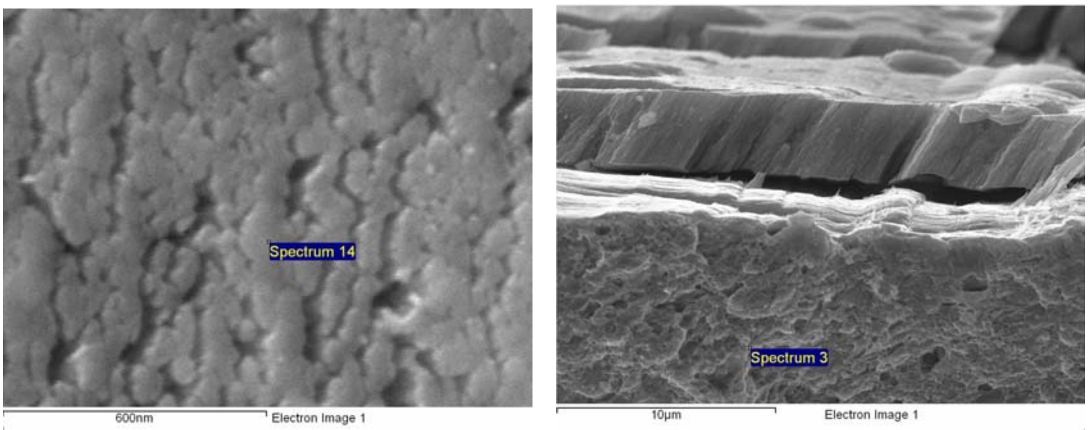

Fig. 18 *The view on the Ti coating surface and the fracture of Ti coating*

Conclusions

Ti coatings created by activated evaporation by electron beam physical vapour deposition method – EBPVD with different position of samples in the vacuum chamber. The following results were obtained:

- Thickness of the Ti coating was at the interval from 5,9 to 7,93 μm in dependence on the distance of the substrate from the axis of the vacuum chamber – it indicates that the temperature of the substrate during deposition 200 °C is enough.
- Hardness of the Ti coatings was from 3,65 GPa to 128,53 GPa, Young´s modulus was from 8 GPa to 53 GPa. Measured hardness values and Young´s modulus values of the Ti layers in comparison publicised data are lower. It is caused by the structure of the layers, which grow in impact angles of Ti atoms smaller than 90° and growth of the layer at lower temperatures. It also can be caused by structure of Ti coating with high roughness (R_a = 0,42 μm) of the surface (see Fig. 18).
- Adhesion of the Ti coating was from 13 N to 33 N, that is lower than publicized ones, but the maximum values are considered as a suitable extent of adhesion.
- Measured wear of the Ti coating was 7,564. 10^{-3} g and the counterpart (steel ball) 8,433. 10^{-3} g. The friction coefficient was μ = 0.4 (track 50 m) and maximum value μ = 0.9 (track above 55 m to 90 m). In comparison with publicised data the results are very good. In this case an impact angles of Ti atoms smaller than 90° is advantage compared an impact angle of 90° and the method magnetron sputtering.

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