PROPERTIES OF Ti COATINGS EVAPORATED ON STEEL SUBSTTRATE SITUATED IN VACUUM CHAMBER IN DIFFERENT POSITIONS

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Summary. Mechanical properties such as thickness, hardness, adhesion and roughness of the Ti coating were evaluated by authors. There were achieved values of thickness, hardness, and adhesion equal to 2.8 and 2.6 µm; 2.4 and 4.9 GPa, 31 N and 20 N, respectively. For evaluation above mentioned properties have been used scanning electron microscopy (SEM), microhardness apparatus and Scratch test, respectively. Ti coating has been activated reactive evaporated (ARE) onto steel substrate situated on different positions toward evaporated Ti.

Keywords: evaporation; Ti coating; thickness, hardness; adhesion

1. INTRODUCTION

Ti based coatings are widely used in industry, biomedical engineering and microelectronics as hard wear resistant layers with good coefficient of friction (COF). On other hand Ti coatings have low hardness, high COF. Also they have good adhesion and suitable thermal expansion which makes them good materials for interlayer used to improve adhesion of the functional layers [1, 7, 10-12]. Properties of Ti coatings have been studied extensively [1-14].

S. Kataria et al. [3, 4] studied selected mechanical properties of Ti thin films deposited by magnetron sputtering PVD technique on D9 steel. The film hardness measured at the surface of the coatings exhibited a value of 2.5 GPa. Adhesion (critical load to failure by delamination) for this coating was evaluated at 2 N. N. Chelliah and S.V. Kailas [5] obtain values of hardness of titanium coating equal to 1.65 GPa. B. Bhushan and B. K. Gupta in [6] and T. Jamal et al. [7] presented values of hardness of Ti coating equal to 2.25 GPa. N. Randall in [8, 9] shows values of of Ti coating equal to 16 GPa. K. Chu et al. [10] measured of titanium and titanium diboride monolayers and Ti/TiB2 multilayers. They measured hardness of Ti interlayer 12,5GPa. J. Xu et al. [11] obtained hardness of Ti interlayer 8.6 GPa. G.S.Kim et al. [12] presented hardness of Ti layer 8.2 GPa. A.A. Voevodin et al. [13] evaluated design of a Ti/TiC/DLC functionally gradient coating. Ti coating was prepared by a hybrid of magnetron sputtering and pulsed laser deposition. They measured values of hardness of Ti interlayer equal to 4 GPa. K. Miyoshi et al. [14] measured hardness of 50 nm thick Ti interlayer deposited onto AISI 440C stainless steel equal to 4 GPa.

The presented paper focus on the determination of Ti coating properties deposited on steel substrate under the particular angle to the crucible with evaporating Ti. Authors compare measured results to published properties.

2. EXPERIMENTAL DETAILS

Ti coating deposited on steel substrate OKhN3 MFA was evaluated. Chemical composition is: C=0.36 %; Mn=0.5 %; Si=0.25 %; Cr=0.82 %; Ni=2.87%; Mo=0.3 %; V=0.12 %; Cu=0.15 %; s=0.004 % and P=0.014% (from producer fi Matec, Slovakia). Samples had dimensions 30x5x5 mm. Titanium was deposited by evaporation using the experimental device ZIP-12 (Fig. 1). All coatings were deposited within one process cycle. Power of electron gun was 2.5 kW, vacuum chamber pressure 0.01 Pa, cathode current was 0.2 A, ARE electrode voltage was 240 V and coating deposition time was 30 min. Before deposition, substrates were cleaned by ultrasound 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 = 10 min. Distance between the devaporating material and sample surface (cathode) was 360 mm, distance between the samples and cathode axis was 100 and 140 mm (Fig. 2).

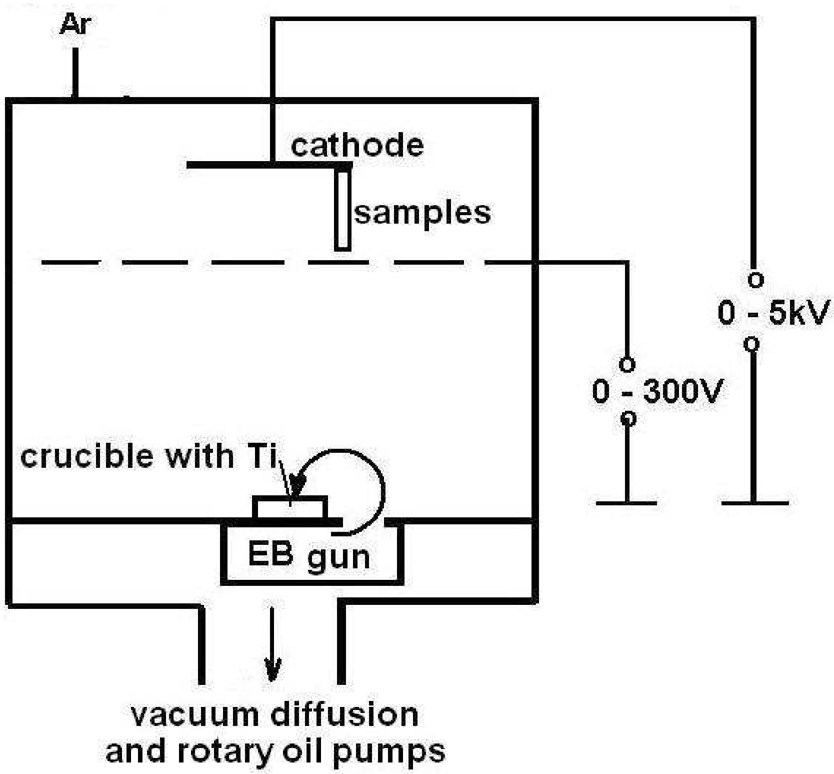

Figure 1 The apparatus for activated evaporation using electron beam (EB PVD), schematically

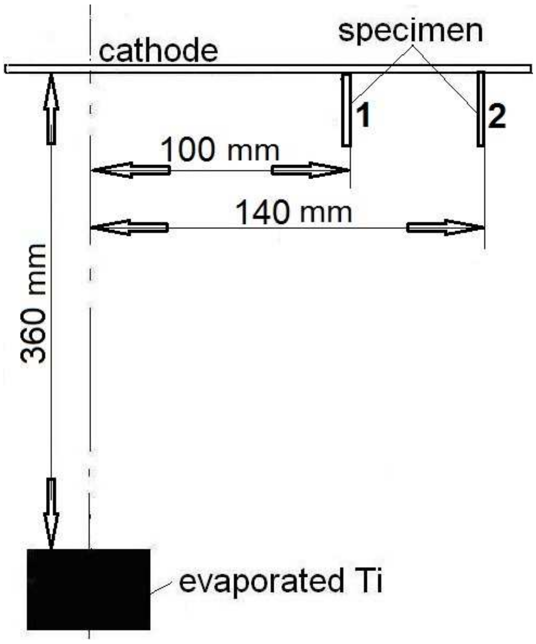

Figure 2 Locations of samples (1 and 2) inside the vacuum chamber, schematically

The adhesion of the Ti coatings on steel substrate was examined using Scratch test [4]. The test was made by moving a Rockwell diamond tip to the specimen surface with a gradually increasing loading force up to 120 N. The evaluation of the adhesion was done by optical microscope according to the amount of cracks and delamination around the scratch [4].
Thickness of the Ti coatings were determined by scanning electron microscope Jeol 7000F. The evaluation of the ***hardness*** was carried out using a CSM Instruments tester. Measurements were made using Berkovich indenter with sinus load mode at frequency of loading 20 Hz. The peak load was 0.07 N. The used penetration depths were in all cases lower than 10% of the layer thickness.

3. RESULTS AND DISCUSSION

Two samples located in the vacuum chamber (Fig. 2) were evaluated. Thickness of Ti coating was measured: 2.8 µm (sample 1) and 2.6 µm (2). It is obvious an unequal angle between deposited surface and columnar grains of surface. It is not equal 90°. The inequality is caused by deposited surface position towards stream direction of deposited Ti particles. Unequal thickness can be caused by different distance from surface of evaporating Ti. Chemical composition is similar to evaporating Ti (Fig. 5). ***Adhesion*** was evaluated using Scratch test, in the direction of columnar grains slope of coating. Measured values are following: 31 N (Fig. 6) (sample 1) and 20 N (sample 2). From the application point of view, value 30 N is sufficient mainly because of low deposition temperature 200 °C. Because of low deposition temperature it can be assumed that for temperatures above 350°C the adhesion values can grow to 50 N and more, which is considered to be satisfactory.

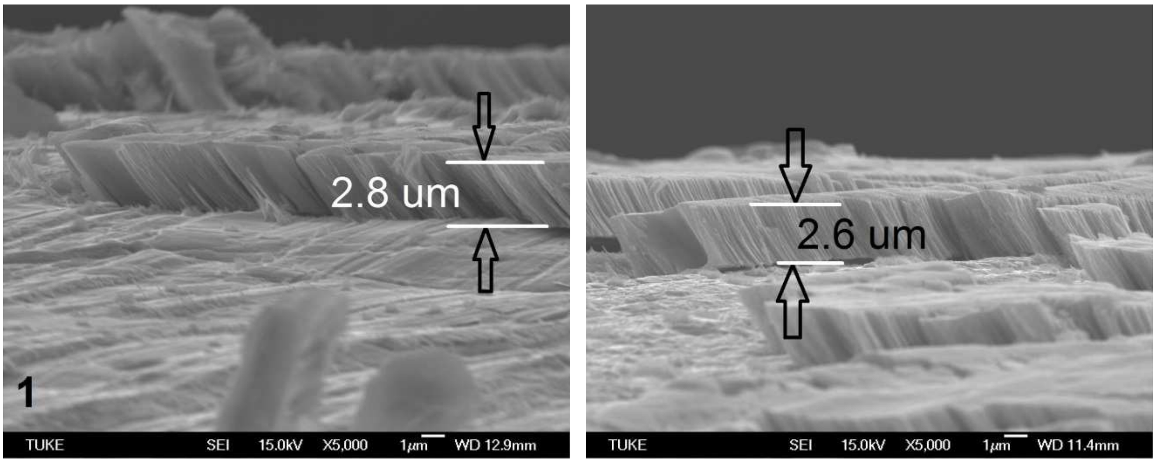

Figure 3 Thickness of Ti coating: (1) 2.8 μm and (2) 2.6 μm

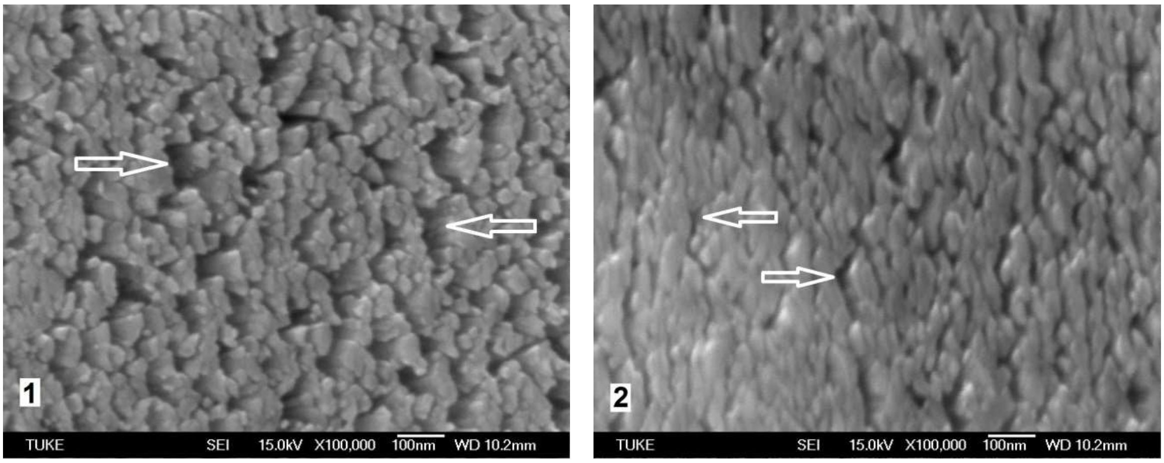

Figure 4 Front view of Ti coating with gaps (see arrows): (1) and (2)

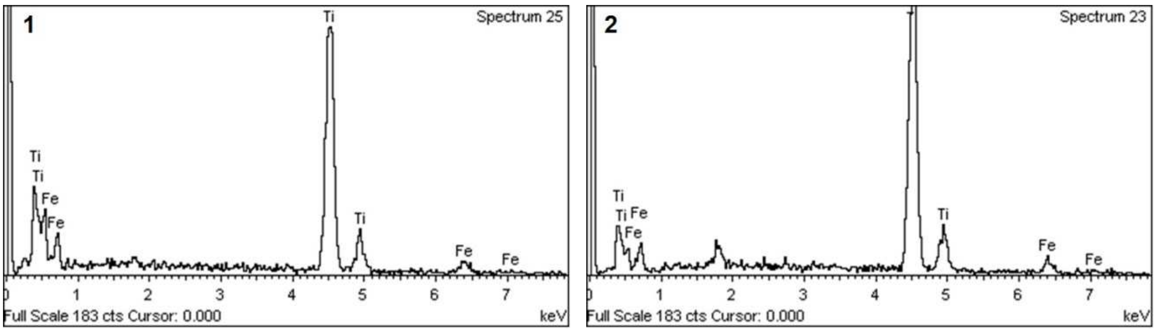

Figure 5 Chemical composition (EDS) of Ti coatings: sample 1 and 2

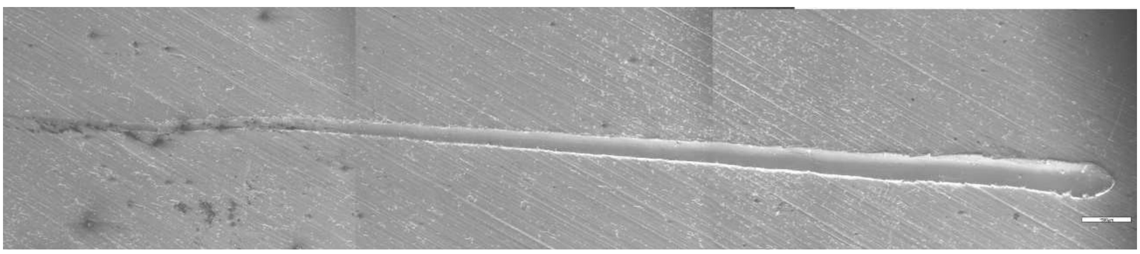

Figure 6 Scratch after Scratch test, coating on the sample 1, adhesion 31 N, total length of scratch 12 mm

Hardness of evaluated Ti coatings was 2.4 GPa (sample 1) and 4.9 GPa (sample 2). Measurements were carried out on equipment by CSM Instruments according to the standard ISO 14577. Each value is an average from 10 measurements. For the hardness of the Ti coating of booth samples is reported the maximum of measured values. Presented difference is caused by many gaps on the deposited surface. Coating 1 (Fig. 4) contains many gaps with width up to 70 nm and length up to 150 nm (see arrows). Gap frequency on sample 1 is much higher in comparison to sample 2 (see arrows). Coating 2 achieved 50 % higher hardness in comparison to coating 1. The result is 3 to 4 times higher than in [3-5, 7, 8], twice as high as that in [13, 14], in agreement with [11, 12], 50% lower than in [10] and 100% lower than those reported in [8, 9].

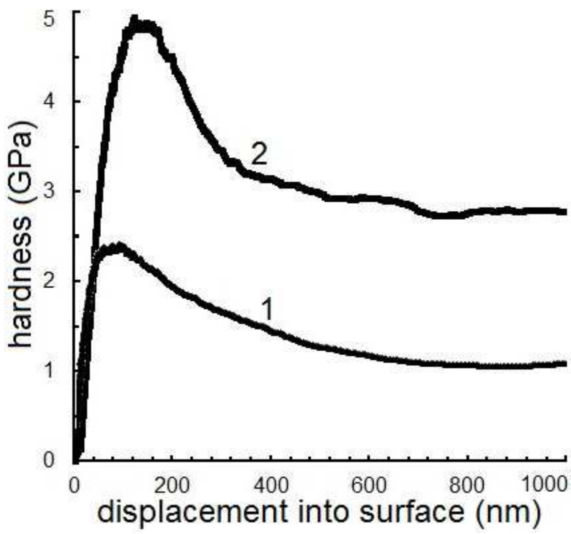

Figure 7 Hardness of Ti coatings: 1 - 2.4 GPa and 2 - 4.9 GPa

4. CONCLUSIONS

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

- following properties of Ti coating deposited onto two substrates made of OKhN3 MFA steel were evaluated: thickness, hardness and adhesion,
- samples were located in vacuum chamber in two different positions towards devaporating Ti, 100 mm a 140 mm from the perpendicular line to devaporated surface. High of samples above the devaporated Ti surface was constant, 360 mm,
- coating width achieved 2.8 µm and 2.6 µm, whereby width decreased proportionally with distance from perpendicular line of devaporated Ti surface,
- vice-versa, hardness increased with the distance from perpendicular line of devaporated Ti surface. Measured values achieved 2.4 GPa and 4.9 GPa, as a consequence of gap frequency on the coated surface. Coating was the more continuous the higher sample position from of devaporated Ti surface,
- adhesion of Ti coating 1 achieved 31 N and Ti coating 2 achieved 20 N,
- two different angles of columnar grain slope of Ti coating were observed as a consequence of unequal of incident angle of evaporated Ti.

Acknowledgement
This work was partially supported by the Slovak Grant Agency VEGA, project number No 1/0432/17. Also, the work was realized within the frame of the project APVV-16-0359.

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