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Fractal nature of surface topography and physical properties of the TiN coatings obtained in the PVD process *

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The paper presents investigation results of the effect of deposition parameters on structure and mechanical properties of the TiN coatings obtained by magnetron sputtering in the vacuum furnace onto the ASP 30 sintered high speed steel. Effect of sputtering parameters on chemical and phase composition, thickness and microhardness were evaluated. The characteristic structure and surface topography of the analysed coatings are presented, the fractal nature of the surface topography was confirmed.

1. INTRODUCTION

In the case of the coatings obtained in the PVD processes numerous physical properties depend on structure and their chemical composition. The coatings are also characterised by specific geometrical properties to describe in which was such concepts as morphology, topography and shape are used. The results of the research indicate that there is a relation between the morphology of the surface of the coatings and the technology used in the process. It is extremely important to define the kind of relation as the morphology of the surface is crucial for such properties of coatings as: roughness parameter, coefficient of friction, hardness and wear resistance [1-5]. The contemporary methods used to describe the topography of the surface of the coatings make it possible to define the relation between the parameters of the technology applied to obtain the coatings, their structure, working properties and their fractal dimension. Thus the description of geometric qualities of the coatings' surfaces obtained in the PVD processes constitutes an important issue of the surface engineering [6-8, 11-14].

The aim of this work is to examine the effect of deposition parameters on the mechanical properties, structure and the fractal dimension of the TiN coatings obtained in the process of magnetron sputtering.

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2. INVESTIGATION PROCEDURE

Experiments were made using the specimens from the ASP 30 sintered high speed steel containing 1.28% C, 4.2% Cr, 5.0% Mo, 6.4% W, 3.1% V, and 8.5% Co. The specimens were heat treated in the salt bath furnaces with austenitizing at the temperature of 1180°C and triple tempering at the temperature of 540°C. After introducing the specimens into the single vacuum chamber with the magnetron built-in for the ion sputtering, the specimens were placed at the distance of 95mm from the magnetron disk. After obtaining the $6\text{-}7{*}10^{-2}$ Pa vacuum, the coating deposition process was carried out with substrate bias equal to 0V, -100V, and –200V respectively. The cleaning process was carried out at the pressure of 25 Pa during 5 minutes with the substrate bias of 900 V in the argon atmosphere. The TiN coatings were developed on the specimens' surfaces. The magnetron disk was made from the titanium alloy containing: 90% Ti, 5.7% Al, 1.4% Cr, and 2.0% Mo.

Evaluation of the phase composition of the obtained coatings was made using the Dron 2.0 X-ray diffractometer, using the cobalt lamp with the voltage of 35 kV and heater current of 7 mA.

The analysis of the texture of the coatings was done using the pole figures' method in which the XRD7 X-ray diffractometer of the Seifert-FPM company was used. The pole figures were obtained by means of the reflection method, in the range of inclination angle of the samples from 0 to 75 with the use of the SMZ7 attachment.

Changes of the chemical composition of the coating components in the direction perpendicular to its surface were evaluated basing on examinations made using the Leco Instruments GDS-750 QDP glow discharge optical emission spectrometer.

Structures of the developed coatings were examined on their transverse fractures on the PHILIPS XL-30 electron scanning microscope.

Coating thickness tests were made using the kalotest method. Thickness tests were made also on the scanning electron microscope to verify the obtained results.

Examinations of the coatings' microhardness were made on the SHIMADZU DUH 202 ultra-microhardness tester.

The tests of the topography of the surfaces of the coatings obtained were carried out in the scanning electron microscope as well as using the method of Atomic Force Microscopy (AFM) in the E Nanoscope of the Digital Instruments. On the basis of the diagrams, the fractal dimension D_s of the analysed surfaces of the coatings was determined. It was done by means of the projective covering method (PCM) (Fig.1) [3,10]. In this method, when *k*th square abcd (a, b, c and d are the four points of the square) with a selected scale of δxδ, the heights of a fracture surface at points a, b, c and d corresponds to h_{ak}, h_{bk}, h_{ck} i h_{dk} (Fig. 1b). The area of rough surface surrounded by points abcd can be approximately calculated by:

$$A_k(\delta)=\frac{1}{2}\left\{\left[\delta^2+(h_{ak}-h_{dk})^2\right]^{\frac{1}{2}}\cdot\left[\delta^2+(h_{dk}-h_{ck})^2\right]^{\frac{1}{2}}+\left[\delta^2+(h_{ak}-h_{bk})^2\right]^{\frac{1}{2}}\cdot\left[\delta^2+(h_{bk}-h_{ck})^2\right]^{\frac{1}{2}}\right\} \quad (1)$$

The entire area of the rough surface under *k*th scale measurement is given by:

$$A(\delta)=\sum_{k=1}^{N(\delta)}A_k(\delta) \quad (2)$$

when N(δ) is the total number of cells with scale of δxδ needed to cover the rough surface.

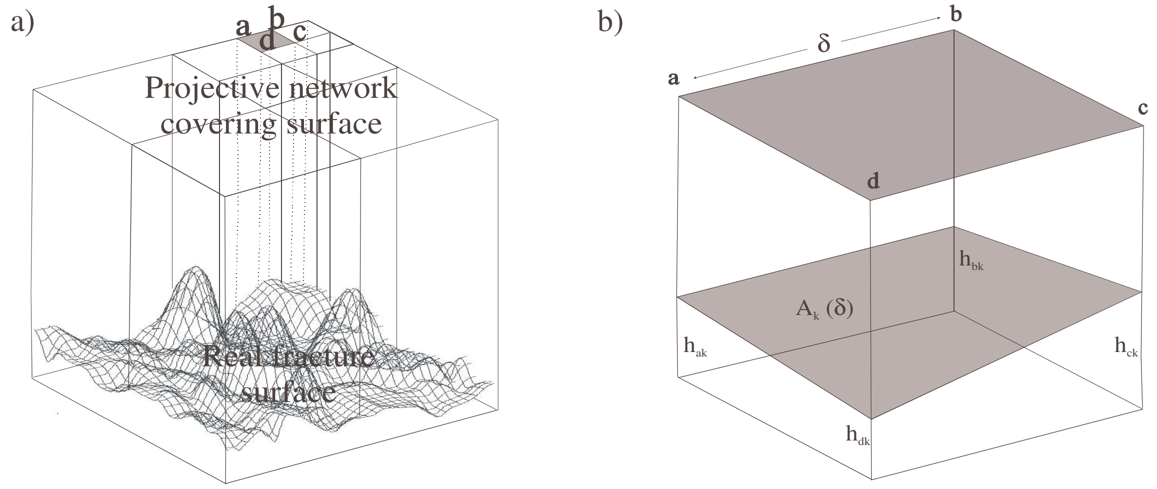


Fig. 1. The projective covering method - a) the division of the plane by means to the square mesh to and their projection on the analysed surface, b) the magnification of *k*th elements of the covering projective surface and the projection on the part of the real fracture surface.

The procedure was repeated with the decreased length of the side (δ) of the mesh. As (δ→0), A(δ) approximates to a real area of the surface as:

$$A\ (\delta) = A_o \delta^{2-Ds} \tag{3}$$

where Ds is the fractal dimension of the analysed surface, $Ds \in [2,3)$ [3, 10, 11]. It means that opposite to the Euclidean surfaces, the dimension of the fractal surface is not still but it rises without any limits together with the rise in the precision of the tests (δ lowering). Slope of the log-log plot A(δ) vs. δ is equal $2\text{-}D_s$. For the real surfaces, the relationship is limited only to a certain range called the fractal range. For the high values of the δ (the upper boundary of fractality) the testing points are placed along the horizontal line, which relates to the $D_s=2$ value. It means that in this scale the analysed surface is seen as flat. For the δ values being to small (because of the testing abilities), the inclination of the straight line (the lowest boundary of the fractality), along which the testing points are placed, decreases to the level of the horizontal line.

3. DISCUSSION OF THE INVESTIGATION RESULTS

Basing on test results conducted on the scanning electron microscope it was found out that the analysed TiN coatings obtained with substrate bias equal to 0V are characterised by the columnar structure, and the coatings obtained with substrate bias equal to –100 and –200V are characterised by the amorphous structure. All analysed coatings are uniformly deposited onto the entire surface and tightly adhere to the substrate material (Fig. 2).

The characteristic endings of the columns which build the coating observed on the surface, are similar to upturned pyramids, cones and craters (Fig. 3).

The results of the X-ray quality phase analysis prove that there are the TiN coatings deposited on the high-speed steel of the ASP type. However, the magnetron disk was not made of the pure titanium but from the alloy containing 90% Ti, 5,7% Al, 1,4% Cr and 2,0% Mo, and therefore the identified phase TiN may be denoted as (Ti, Al, Cr, Mo, Fe, Si)N which was confirmed by means of the glow discharge spectrometer. The analysis made by

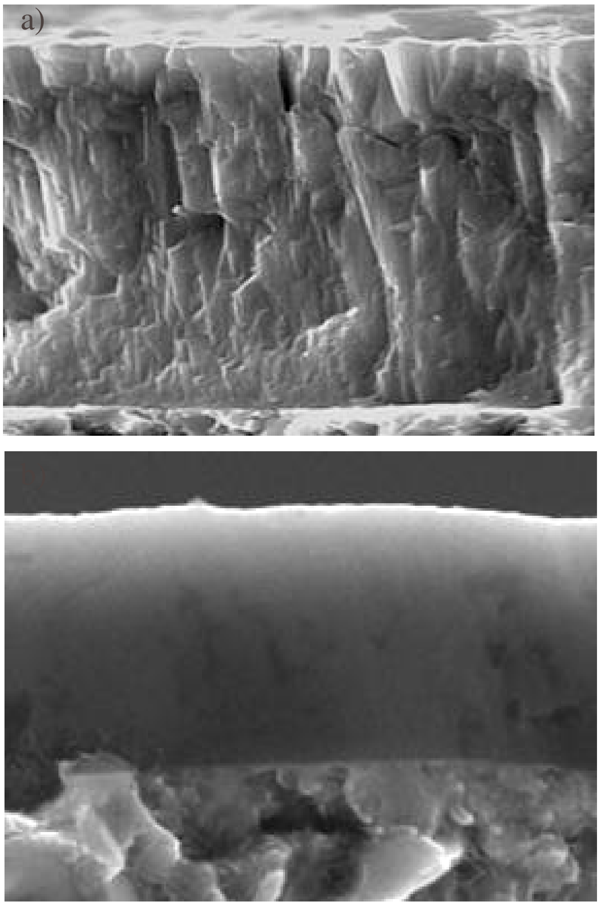


Fig. 2. Fracture of the TiN coating (coating deposition conditions: a) substrate bias – 0V, b) substrate bias –200V).

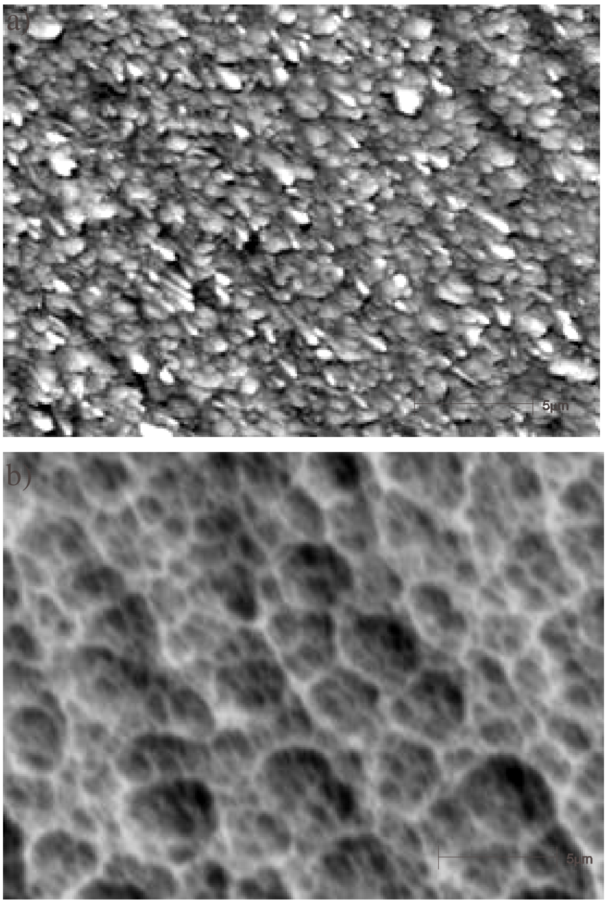


Fig. 3. Surface topography of the TiN coating (coating deposition conditions: a) substrate bias – 0V, b) substrate bias – 200V).

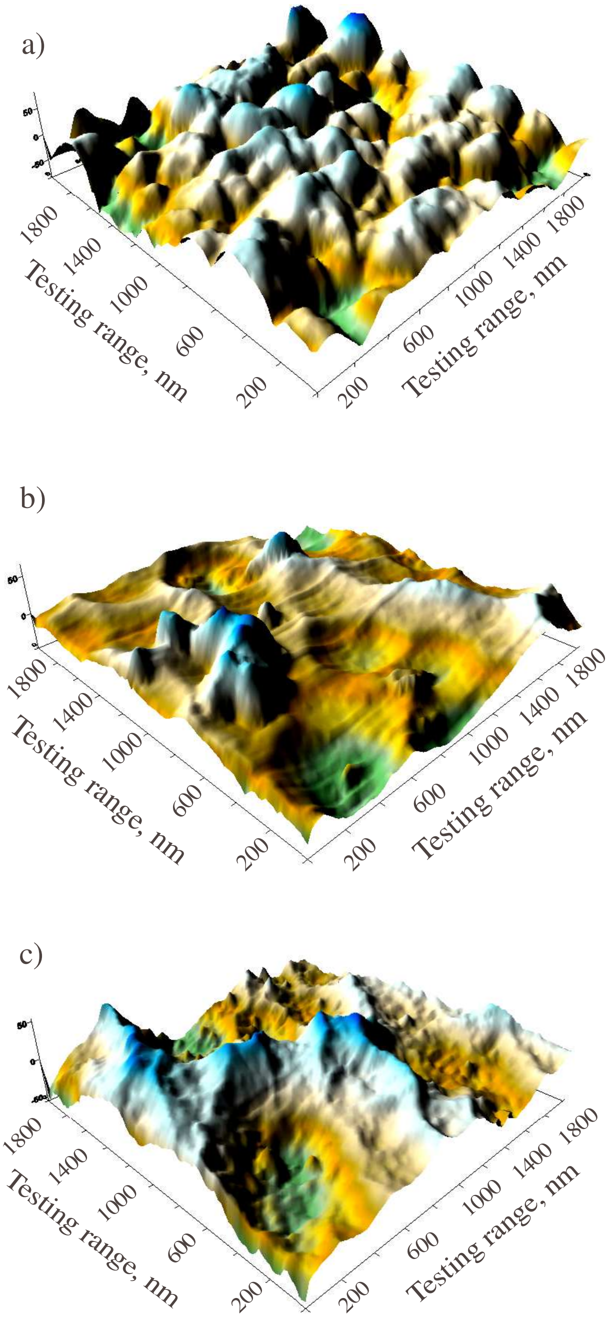
means of the same device proved the existence of Cr, Mo, W and Fe in the coatings tested. The total concentration of these elements does not exceed 2% atomically depending on the conditions in which the coatings were deposited.

The influence of the substrate bias on the thickness, hardness and atomic concentration of the Ti, N and Al in the analysed coatings is presented in Table 1.

On the basis of texture tests it was stated that the analysed coatings are characterised by the axis texture, differential for the surface of coatings' increase. The surface polarization change from 0 [V] to -100, - 200 [V] caused change in the crystallographic orientation of the

Table 1. Summary of thickness, hardness and Ti, N and Al concentrations in the analysed coatings

Substrate bias [V]	Concentrations of elements atomic, %			Thickness	Hardness
	Ti	N	Al	µm	HV $_{0,05}$
0	41,6	52,3	3,1	4,28	3000
-100	39,9	52,1	5,6	4,08	3100
-200	42,7	52,9	1,5	2,94	3100



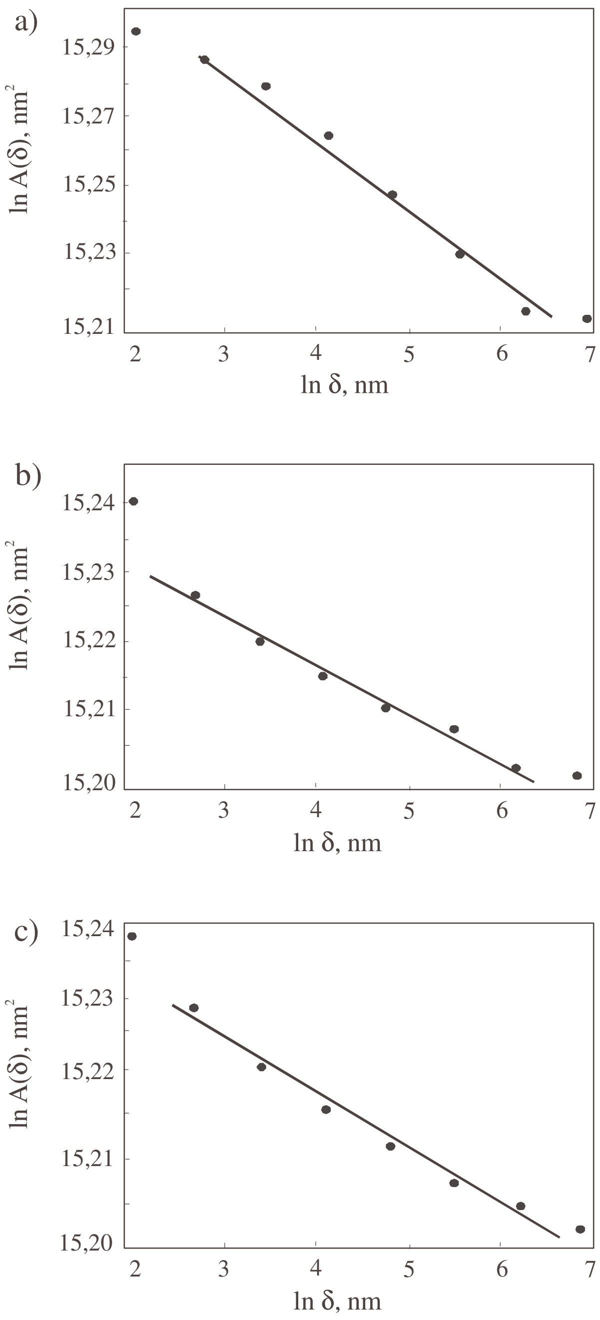


Fig. 4. The picture of topography of the surface of the TiN coatings obtained in the substrate bias a) 0V, b) –100V, c) –200V (the AFM microscope, testing range – 2μm).

Fig. 5. The estimation of fractal dimension of the TiN coating obtained in the substrate bias a) 0V, b) –100V, c) –200V (testing range – 2μm).

investigated coatings from <111> in case of coatings obtained with substrate bias equal to 0 [V] to <110> in case of coatings obtained with substrate bias equal to – 100 [V] and – 200 [V] [1,2].

The fractal dimension was determined by means of the projective covering method. In order to calculate the fractal dimension the images of the topography of the analysed coatings were used. The images were obtained in the AFM (Fig. 4). Each image constitutes matrix of heights of 512x512. The following values of the δ amounted respectively – the scanning range divided by n, where n=2,3…8. The values A(δ) defined in the test were placed on the log-log plot (Fig. 5). The fractal range was defined and the straight line was adjusted by means of the least squares' method. The slope of the straight line was used to determine the D_s value. The fractal dimension was calculated as the weighted average value of three testing results for every specimen. The results confirm the fractal character of the analysed coatings. The lowest values of the fractal dimension – 2,0061 and 2,0066, were obtained for the coatings evaporated with substrate bias equal to -100 [V] and – 200 [V] respectively. The highest value of the fractal dimension – 2,0214 was obtained for the coatings evaporated with substrate bias equal to 0 [V]. The results of the calculations of fractal dimension are connected with the figure of surface topography of the analysed coatings.

4. SUMMARY

Basing on the investigations carried out, it was found out, that the highest value of the fractal dimension – 2,0214±0,001 was obtained for the coatings evaporated with substrate bias equal to 0 [V], and the lowest value of the fractal dimension - 2,0061±0,0019 and 2,0066±0,0021 was obtained for the coatings evaporated with substrate bias equal to -100 [V] and – 200 [V] respectively. The change of fractal dimension and substrate bias from 0 [V] to -100, - 200 [V] is connected with the change in the crystallographic orientation from <111> in case of coatings obtained with substrate bias equal to 0 [V] to <110> in case of coatings obtained with substrate bias equal to – 100 [V] and – 200 [V].

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