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Structure, physical properties and fractal character of surface topography of the Ti+TiC coatings on sintered high speed steel

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Abstract: The paper presents investigation results of the effect of deposition parameters on chemical and phase composition, structure, texture, thickness, micro-hardness and fractal dimension of surface topography of the two-layer Ti+TiC coatings obtained by magnetron sputtering in the vacuum furnace onto the ASP 30 sintered high speed steel.

Keywords: PVD coatings, fractal geometry, texture

1. INTRODUCTION

In the case of the coatings obtained in the PVD processes numerous physical qualities depend on the structure and the chemical composition [1]. The coatings are also characterised by specific geometric properties to describe which 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 [2, 3]. 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, usable properties and their fractal dimension [4-12]. Thus the description of geometric qualities of the coatings' surfaces obtained in the PVD processes constitutes an important issue of the surface engineering.

2. EXPERIMENTAL 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) [3,9]. 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}. 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. 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:

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

where Ds is the fractal dimension of the analysed surface, Ds∈[2,3) [3, 7-9]. 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-D_s. For the real surfaces, the relationship is limited only to a

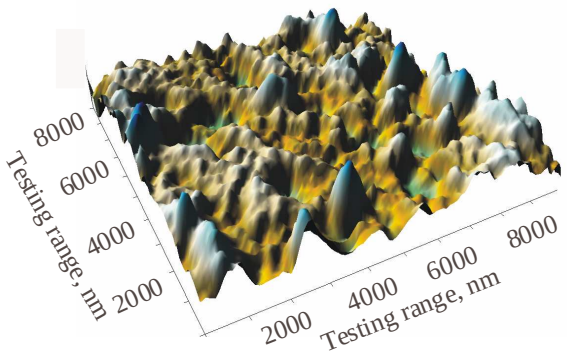

Fig. 1 The picture of topography of the surface of the Ti+TiC coatings obtained in the 540°C (the AFM microscope, testing range – 10 μm).

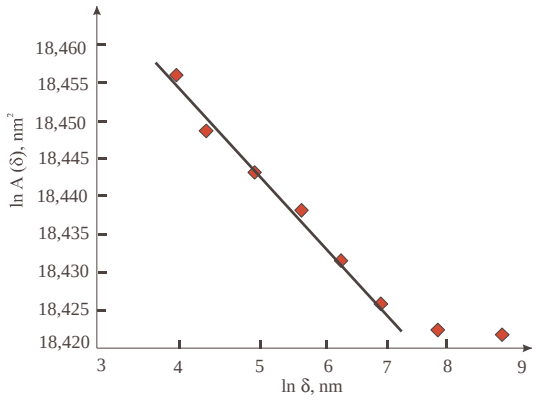

Fig. 2 The estimation of fractal dimension of the surface of the Ti+TiC coating obtained in the 540°C (testing range – 10μm).

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. RESULTS AND DISCUSSION

On the basis of tests conducted in the scanning electron microscope it was stated that the analysed Ti+TC coatings are characterised by the amorphous structure, they are evenly deposited on the whole surface and they strictly adhere to the substrate material. The characteristic endings of the columns which build the coating observed on the surface, are similar to upturned pyramids, cones and craters. The results of the x-ray quality phase analysis prove that there are the Ti+TiC coatings deposited on the high-speed steel of the ASP type. However, the shield of magnetron 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 TiC may be denoted as (Ti, Al, Cr, Mo, Fe, Si)C which was confirmed by means of the glow discharge spectrometer. The influence of the process temperature on the atomic concentration of the Ti, C, N and Al in the analysed coatings is presented in Table 1. The analysis made by 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. Basing on texture examination it was found out that all analysed Ti+TiC coatings are characterised by axis texture <110>. The texture of coatings obtained in 460 is definitely more weak than for coatings obtained in 500 and 540.

It was found out basing on the Ti+TiC coatings micro-hardness tests that their results should be connected with the chemical compositions of the analysed coatings. The total C and Al concentration increase in coatings is accompanied by hardness increase.

The fractal dimension of the investigated coatings was determined using the projective covering method. Surface topography images of the analysed coatings obtained on the AFM

microscope and saved in a text file as a 512 x 512 matrix of measurement points were used for its determining (Fig. 1). The successive δ values were equal to n, where n=2,3,...8. The determined values $A(\delta)$ were presented in the bilogarithmic plots (Fig. 2). Having determined the fractality range, the straight line was fitted using the least squares method, whose slope was employed to determine the D_s value. The approximate values of the fractal dimension were obtained for various scanning ranges. Presented plots confirm the fractal character of the analysed coatings in a broad range of magnification (2, 5 and 10 μm). The fractal dimension was calculated as an average basing on measurements obtained for various scanning ranges. The highest value of the fractal dimension D_s – 2.017 ± 0.0019 and 2.0237 ± 0.0018 was obtained for the Ti+TiC coatings evaporated in 540ºC and 500ºC. The lowest value – 2.011 ± 0.0023 was obtained for the coatings vaporized in 460ºC. The results of the fractal dimension tests correspond with the results of the hardness parameter tests and texture analysis. Increase of <110> orientation is accompanied by increase of fractal dimension value.

4. CONCLUSION

The research done proved the fractal character of the surface topography of the analysed coatings. The highest value of the fractal dimension was obtained for the Ti+TiC coatings deposited at 540°C and its values is 2.037. The lowest value of the fractal dimension – 2.011 was obtained for coatings deposited at 460°C. The results of the fractal dimension tests correspond with the results of the hardness parameter tests and texture analysis. Increase of <110> orientation is accompanied by increase of fractal dimension and hardness of the analysed coatings.

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REFERENCES

1. Kwaśny W., Dobrzański L.A., Inżynieria Materiałowa, Vol. 3-140, (2004) 607.
2. Dobrzański L.A., Kwaśny w., Inżynieria Materiałowa, Vol. 6-137, (2003) 456.
3. Kwaśny W., Dobrzański L.A., Pawlyta M., Żak J., Proceedings of the MTM, Modern Technologies and Machines, 2-4 October, Cluj-Napoca, Romania (2003) 279
4. Mandelbrot B.B., Form, New York, Freeman 1983.
5. Heping X., Wang J.: International Journal of Solids and Structures (1999) 36
6. Chaudhari, S., Ching-Cher Sanders Y., Shyi-Long L., Applied Surface Science 238 (2004) 513.
7. Seung-Bok L., Su-Il P., Journal of Electroanalytical Chemistry 556 (2003) 75
8. Shek C.H., Lin G.M., Lee K.L., Lai J.K.L.: Journal of Non-Crystalline Solid 224 (1998) 244
9. Hou S.M., Ouyang M., Chen H.F., Liu W.M., Xue Z.Q., Wu Q.D., Zhang H.X., Gao H.J., Pang S.JThin Solid Films 315 (1988) 322
10. Zhiwen Ch., Shuyuan Z., Shun T., Mingliang T., Jianguo H., Yuheng Z.: Thin Solid Films 322 (1998) 194.
11. Tanaka N., Kawahara M.: Materials Science and Engineering A 312 (2001) 25
12. Chen Z.-W., Zhang S.-Y., Tan S., Hou J.-G.: Materials Research Bulletin 37 (2002) 825