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THE INFLUENCE OF DEPOSITION CONDITIONS ON STRUCTURE OF PVD COATINGS OBTAINED IN THE MAGNETRON PROCESS

WPŁYW PARAMETRÓW NANOSZENIA NA STRUKTURĘ POWŁOK OTRZYMANYCH W MAGNETRONOWYM PROCESIE PVD

Abstract

This paper presents the influence of deposition conditions on texture and stress results of TiN coatings deposited by using the PVD magnetron process. Modification of parameters was related to the changes of temperature and substrate potential. The article presents the results of an analysis of texture and stress measurement obtained by X-ray methods and the results of microhardness measurement.

Keywords: *PVD coatings, stress analysis, structure and texture, X-ray analysis*

Streszczenie

W pracy przedstawiono wpływ parametrów nanoszenia na teksturę i naprężenia powłok TiN uzyskanych w magnetronowym procesie PVD. Modyfikacja parametrów dotyczyła zmiany temperatury procesu oraz polaryzacji podłoża. W artykule przedstawiono wyniki analizy tekstury i pomiaru naprężeń uzyskane metodami rentgenowskimi oraz wyniki pomiaru mikrotwardości.

Słowa kluczowe: *powłoki PVD, analiza naprężeń, struktura i tekstura, analiza rentgenowska*

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1. Introduction

In response to the growing requirements for modern engineering materials, one can observe an increased intensity of research that is moving towards developing materials and coatings with the best properties. Nowadays, one of the most popular groups of coating deposition methods is the physical vapor deposition (PVD). This method allows the preparation of many types of coatings, both mono and composed of several elements or phases. With the advancement of science many substitutes of the PVD process were developed, thereby modifying its usability. The aim of this study was to investigate the influence of the deposition condition on the structure of PVD coatings obtained in the magnetron process. [1, 2, 4].

2. Material for study and research methodology

Experiments were made on samples from the PM-HS6-5-3-8 sintered high speed steel contained 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 salt bath furnaces with austenitizing at the temperature of 1180°C and triple tempering at the temperature of 540°C. The specimens were loaded into a single vacuum chamber with a magnetron designed for the ion sputtering, then were placed at the distance of 70 mm from magnetron disk. After obtaining the $6–7 \cdot 10^{-2}$ Pa vacuum the coating deposition process was carried out in 460, 500 i 540°C with the substrate potential equal to 0 V and in 500°C with the substrate potential equal to –100 V and –200 V. The cleaning process was carried out at the pressure of 25 Pa during 5 minutes, with the matrix potential of 900 V in the argon atmosphere. The TiN layer was deposited for 60 minutes under conditions given in Table 1. The magnetron disk was made from titanium alloy containing: 90% Ti, 5,7% Al, 1,4% Cr i 2,0% Mo [1, 2, 4].

Table 1

Coating deposition process parameters

Process temperature [°C]	Substrate potential [V]	Chamber pressure [Pa]	Magnetron voltage, U [V]	Magnetron current, I [A]
460	0	$5{,}1 \cdot 10^{-1}$	360	7
500	0	$5{,}1 \cdot 10^{-1}$	360	
	–100	$5{,}2 \cdot 10^{-1}$	350	
	–200	$5{,}1 \cdot 10^{-1}$	350	
540	0	$5{,}1 \cdot 10^{-1}$	360	

Evaluation of phase composition of the obtained coatings was made using PANalytical X'Pert PRO diffraction system, using filtered radiation from the cobalt lamp with a voltage of 40 kV and heater current of 30 mA. The X-ray phase analysis of the investigated coatings was conducted in the Bragg-Brentano geometry and in grazing incidence geometry (primary beam angle – 2°) using appropriately the PIXcel 3D detector and a proportional detector.

Measurements were made within the 2Θ angle range from 40 to 100°. In order to analyse the texture of the created coatings, three pole figures were measured for each analyzed sample using a reflection method employing Euler's circle of diameter 187 mm in a range of samples inclination angle from 0 to 80°. The orientation distribution function (FRO) was conducted by the ADC method using iterative operator in LaboTex 3.0 [3]. Stress analysis was made using the $\sin^2\psi$ technique in the X'Pert Stress software [1, 2, 5].

3. The results

The results of X-ray phase composition analysis confirmed the existence of the expected layer. Figure 1 shows the diffraction pattern of the analyzed coating in Bragg – Brentano and grazing incidence geometry.

a) b)

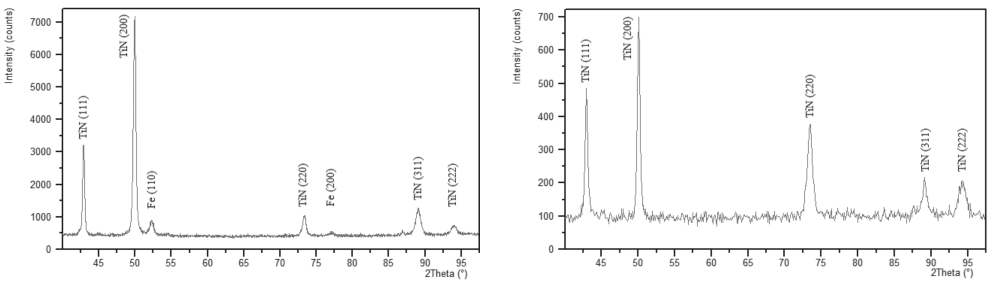

Fig. 1. Diffraction pattern of the sintered high speed steel PM HS-6-5-3-8 with the TiN coating performed in: a) Bragg – Brentano geometry, b) grazing incidence angle geometry (substrate potential 0 V)

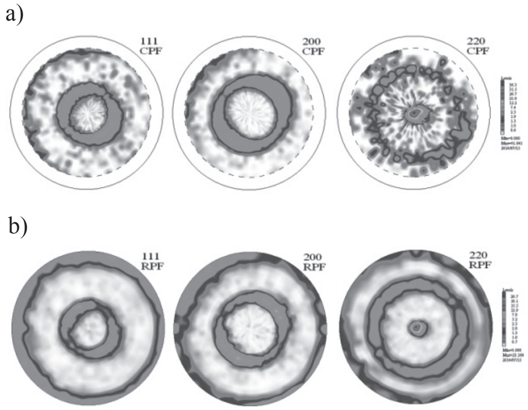

Fig. 2. Pole figures (111), (200) and (220) TiN coating deposited onto the PM HS6-5-3-8 sintered high speed steel obtained by magnetron sputtering: a) experimental, b) calculated by FRO (substrate potential –100 V)

Texture analysis of the examined coatings was conducted based on 3 experimental pole figures (Fig. 2a). FRO of the analyzed materials was shown in figure 3 and pole figures calculated by FRO in Figure 2b. The analysis of pole figures indicates the occurrence of axial component texture <100> for layers made with substrate potential 0V. A negative substrate potential caused change orientation from <100> to <110>. Particular results of texture analysis are shown in Table 2.

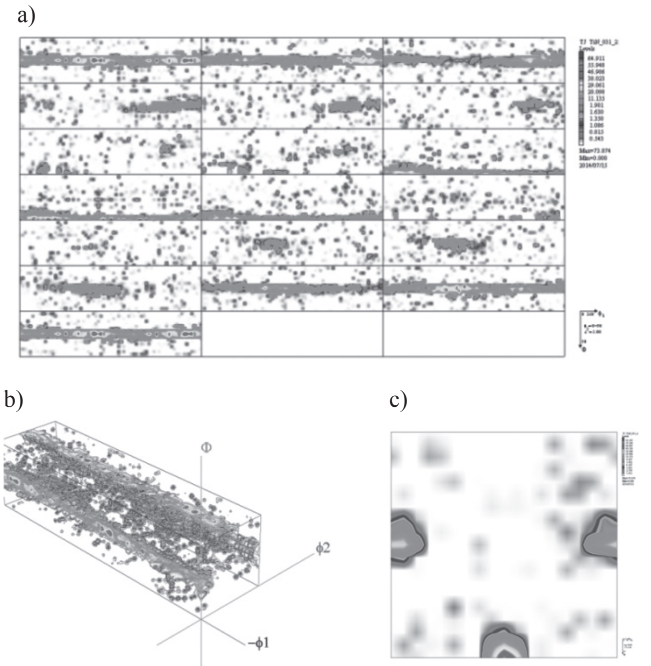

Fig. 3. FRO of TiN coating deposited onto the PM HS6-5-3-8 sintered high speed steel by magnetron sputtering: a) section φ_2, b) FRO, 3D view, c) section φ_1 (substrate potential –100 V)

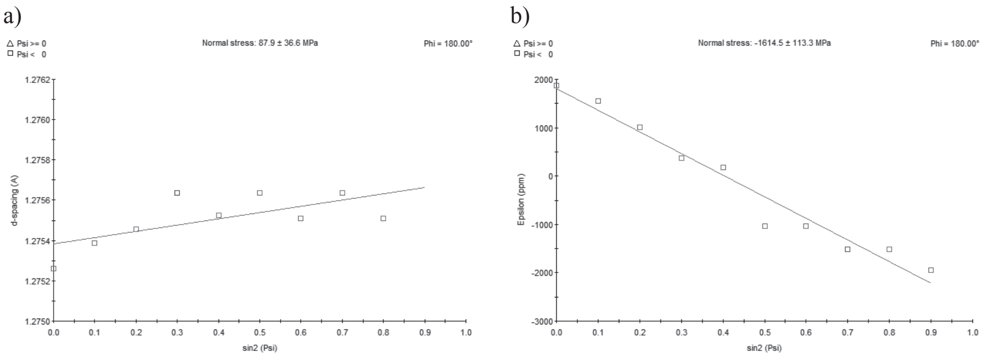

Fig. 4. Stress measurement results of TiN layer deposited in: a) substrate potential 0V, b) substrate potential –100 V

Stress calculated by the $\sin^2\psi$ method (Fig. 4) has shown that coatings made in the negative substrate potential have severe compressive stress. It was unable to determine the stress results for coatings made in the substrate potential equal –200 V due to presence of a strong texture <110>. Particular results of stress analysis and microhardness measurements are shown in Table 2.

Table 2

Results of the texture and stress analysis and microhardness measurements of TiN layers obtained in the magnetron sputtering

Process temperature [°C]	Substrate potential [V]	Texture (type / share)	Stress [MPa]	Microhardness [$HV_{0.1}$]
460	0	<100> / 37%	411.6±77.8	1817
500	0	<100> / 35%	87.9±36.6	1785
	–100	<110> / 31%	–1614.5±113.3	2661
	–200	<110> / 45%	–	1977
540	0	<100> / 40%	–592.7±79.6	1983

4. Conclusions

Based on the experimental results the following conclusions were made:

1. Based on the stress measurements, it was found that the negative substrate potential affects the growth of compressive stress of coatings. For the substrate potential of –100 V stress amounted to –1614.5 ± 113.3 MPa;
2. Based on the texture analysis, it was found that the negative substrate potential causes a change in the orientation of <100> to <110>;
3. Among the tested coatings, coatings deposited at 500°C with substrate potential of –100 V had the biggest microhardness 2661$HV_{0.1}$ had; these coatings had the highest compressive stress, too.

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