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Effect of the substrate bias on structure and properties of the Ti+Ti(C,N) 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 two-layer Ti+Ti(C,N) 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 compositions, thickness, micro-hardness, Young's modulus and adhesion of coatings to the substrate material was evaluated. The characteristic structure and surface topography of the analysed coatings are presented.

1. INTRODUCTION

The research topics connected with deposition of coatings on tool materials feature some of the most important trends in the surface engineering development, ensuring development of coatings with favourable working properties like mechanical properties and wear resistance [1-3]. Applying the new working properties to commonly used materials is obtained often by deposition of coatings with the PVD techniques, which – when used effectively – make it possible to improve significantly the working properties of tools made from high steels, whose technology is fully developed. One of them is magnetron sputtering that is used most often to obtain very hard coatings, e.g., AlN, TiN, Ti(C,N) and TiC [4,5].

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 were into the single vacuum chamber with the magnetron built-in for the ion sputtering, the specimens were placed at three different distances from the magnetron disk, equal to 70, 95, and 125mm

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Table 1
Coating deposition process parameters

Coating type	Working atmosphere	Substrate bias [V]	Pressure in the chamber [Pa]	U Magnetron voltage [V]	I Magnetron current [A]
Ti	Ar	0	$8*10^{-1}$	400	5
Ti(C,N)	$50\%N_2+50\%CH_4$	0	$5,6*10^{-1}$	420	5
Ti	Ar	-100	$8*10^{-1}$	400	5
Ti(C,N)	$50\%N_2+50\%CH_4$	-100	$5,6*10^{-1}$	410	6
Ti	Ar	-200	$8*10^{-1}$	400	5
Ti(C,N)	$50\%N_2+50\%CH_4$	-200	$5,6*10^{-1}$	410	6

respectively. 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 matrix potential of 900 V in the argon atmosphere. The Ti+Ti(C,N) coatings were developed on the specimens' surfaces. The Ti interlayer was deposited during 6 minutes and afterwards the next layer was deposited during 60 minutes in conditions given in Table 1. 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 filtered cobalt lamp rays with the voltage of 35 kV and heater current of 7 mA.

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. Tests were made with the 0.05 N load, making 6 impressions for each examined specimen, so that the impressions' depths were smaller than 1/10 thickness of the deposited coatings, thus eliminating to a significant extent the influence of substrate on the obtained measurement results.

3. DISCUSSION OF THE INVESTIGATION RESULTS

It was found out basing on examinations made on the scanning electron microscope that the analysed Ti+Ti(C,N) coatings are uniformly deposited onto the entire surface and tightly adhere to the substrate material demonstrating a glassy structure [3]. The examined structure of the coatings corresponds to the I zone of the Thornton's model [6]. No crystalline structure elements were revealed in examinations of these coatings on the scanning microscope, even at very large magnifications. Characteristic fracture surface of the Ti+Ti(C,N) coating is presented in Figure 1 and its corresponding surface topography image is presented in Figure 2.

Results of the qualitative X-ray phase analysis confirm that the Ti+Ti(C,N) coatings were developed on the investigated ASP type high speed steel (Fig.3). Taking into account that the

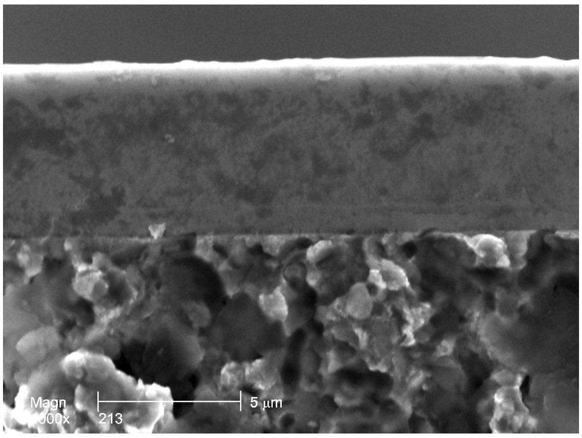

Figure 1. Fracture of the Ti+T(C,N) coating (coating deposition conditions: substrate bias –200V, distance of specimens from the magnetron disk 95mm).

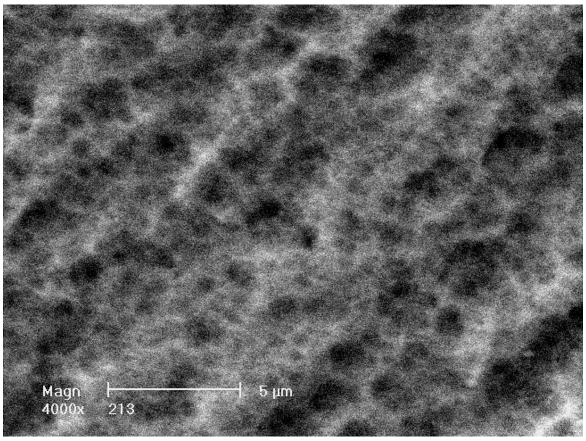

Figure 2. Surface topography of the Ti+Ti(C,N) coating (coating deposition conditions: substrate bias–200V, distance of specimens from the magnetron disk 95mm).

magnetron shield was not made from pure titanium but from its alloy containing: 90% Ti, 5.7% Al, 1.4% Cr and 2.0% Mo, the revealed Ti(C,N) phase could be denoted as (Ti, Al, Cr, Mo, Fe, Si)(C,N), which was confirmed by investigations with EDS-method on substrate surface (Fig.4) and by examinations in the glow discharge optical emission spectrometer. Transverse section profiles of the analysed coatings (Fig.5) made basing on the examination results (GDOS) indicate that three zones occur in them: the external zone, intermediate, and the diffusion zone between the coating and substrate material, whose width is dependant on substrate material and position of specimens in respect to the magnetron disk [5].

The substrate bias effect on the Ti, C, N, and Al atomic concentrations in the analysed coatings is presented in Table 2. Change of the substrate bias to the negative values causes increase of N concentration and decrease of Ti concentration. The substrate bias change from 0V to –100V, –200V during the coating deposition process results also in slight changes of C concentration and decrease of Al concentration from 10% atomic for coatings developed at the substrate bias 0V to ca. 1% atomic for specimens obtained at the substrate bias equal to –200V. The chemical composition analysis (Fig. 4 and 5) revealed also presence of Cr, Mo, W, Fe in the investigated coatings, whose total average concentration does not exceed 2% atomic, depending of coating development conditions. Thickness of the analysed coatings is within the 3,4 – 11,38µm range, depending on the distance of specimens from the magnetron disk and substrate bias [1,2]. The first of the analysed factors, independently on the substrate bias, causes that the coating thickness increase occurs with the decreasing distance of specimens from the magnetron disk, because of the greater plasma density at smaller distance.

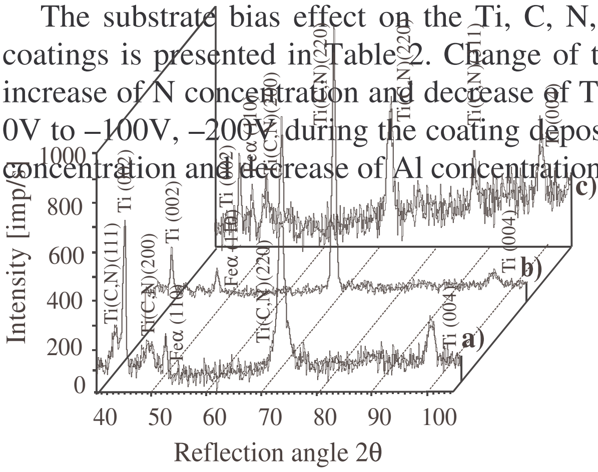

Figure 3. Diffractions patterns of the sintered high speed steel with the Ti+Ti(C,N) coating (coating deposition conditions: substrate bias a) 0V, b) –100V; c) –200V, distance of specimens from the magnetron disk 95mm).

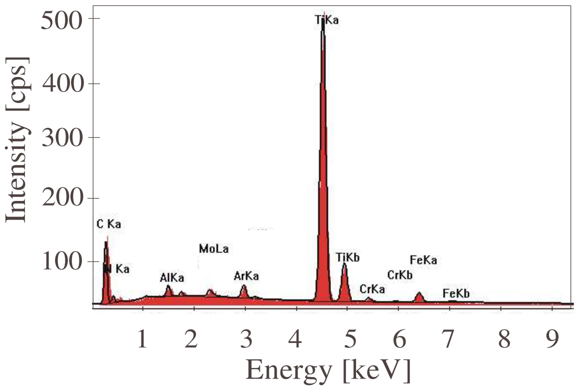

Figure 4. Diagram of energy of dispersed roentgen radiation from surface of Ti+Ti(C,N) coating (condition of coatings' obtaining: substrate bias –200V, distance of specimens from the magnetron disk 95mm).

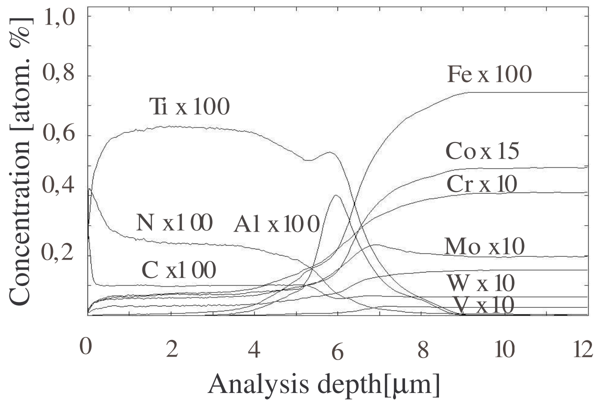

Figure 5. Changes of the Ti+Ti(C,N) coating constituents and substrate material (coating deposition conditions: substrate bias 200V, distance of specimens from the magnetron disk 95mm).

Change of the substrate bias to the negative one results in a slight lowering of the coating thickness increase, therefore, for coatings obtained in different current conditions and the same distances from the magnetron disk similar results were obtained (Fig.6). It was found out, basing on the micro-hardness tests of the Ti+Ti(C,N) coatings, that the highest hardness have the coatings obtained at the substrate bias of –200V. Increase of the negative substrate bias voltage results in the micro-hardness increase of the analysed coatings, regardless of their position in respect to the sputtered disk. The micro-hardness tests results should be connected with the chemical composition of the analysed coatings. The summary N and C concentration increase in coatings is accompanied by hardness increase (Fig.7).

In Figure 8 it is presented the dependence of load and load reduction during penetration in investigated material observed by hardness measurements of the analysed coatings, as basis to mark the Young's modulus of investigated coatings, which value is presented in Figure 9 [7-9].

Table 2
Summary of Ti, C, N and Al concentrations in the analysed coatings, depending on their deposition parameters

Substrate bias [V]	Distance of specimens from the magnetron disk [mm]	Concentrations of elements [atomic, %]							
		Ti		C		N		Al	
		EDS	GDOS	EDS	GDOS	EDS	GDOS	EDS	GDOS
0	70	58	68,1	12	10,3	19	10,6	10	9,5
	95	57	67,5	11,1	10,5	19,8	10,8	10,1	9,7
	125	56,2	66,1	12	10,9	23,7	11,4	7,1	9,9
-100	70	55	64,7	13	10,5	22,9	20,7	2,1	2,5
	95	51	61,8	13	11,1	30,8	23	2,2	2,5
	125	49,6	57,7	15,4	13	32	25,95	2	2,1
-200	70	55,6	66,8	11,2	9,4	30,5	19,8	2,1	2,3
	95	51,6	62,4	12,2	10,9	32,3	24,4	0,9	0,8
	125	49,5	57,1	15,3	12,6	34	25,3	0,2	0,6

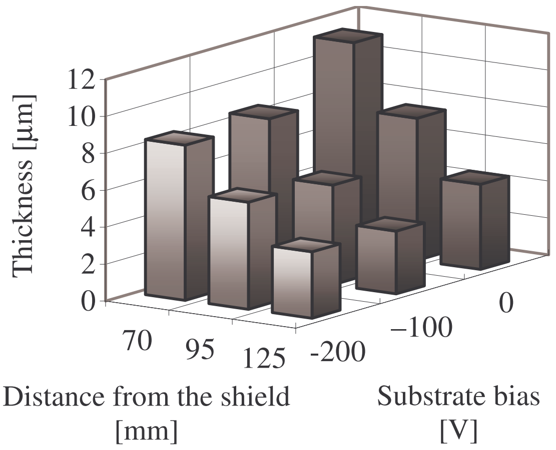

Figure 6. Effect of the substrate bias and distance of specimens from the magnetron disk on thickness of the two-layer Ti+Ti(C,N) coatings.

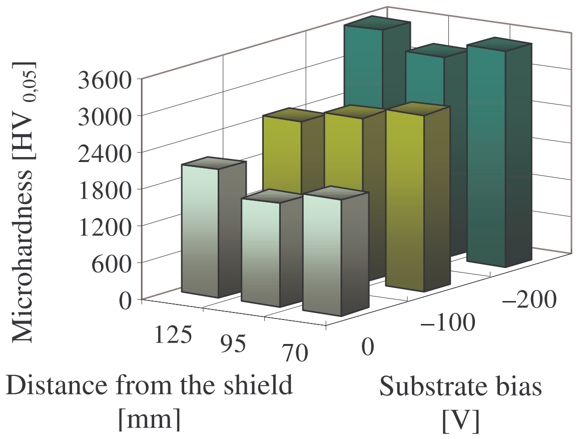

Figure 7. Effect of the substrate bias and distance of specimens from the magnetron disk on microhardness of the two-layer Ti+Ti(C,N) coatings.

Dependence of adhesion of analysed coatings to the substrate material on substrate bias and distance of specimens from the magnetron disk was evaluated with the „scratch-test" method with variable load. It was evaluated the critical load value $L_{c,}$ which allow to determine the strength caused the coatings' damage. The critical load L_c was determined as corresponding to the increase of the acoustic emission increase, signaling beginning of the coating chipping failure. As a result of investigations it was carried out, that the coatings obtained at distance from the magnetron disk of 70mm and at the substrate bias of 0V revealed the highest adhesion to the substrate, whereas the coatings obtained at distance from the magnetron disk of 125mm and at the substrate bias of -200V revealed the lowest adhesion to the substrate. Received results depends mainly on coatings' thickness, what is connected with the distance of specimens from the magnetron disk and substrate bias during obtaining process (Fig.10). The characteristic damage formed during the adhesion investigations with the „scratch-test" method, observed in electron scanning microscope, is presented in Figure 11.

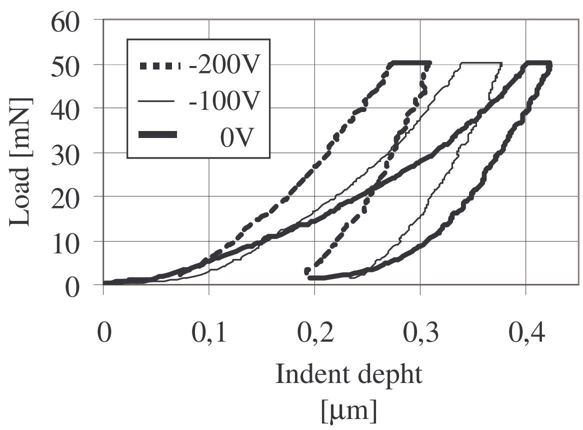

Figure 8. Dependence of load and unload during penetration observed by hardness measurements of the Ti+Ti(C,N) coating obtained at the distance of 95mm.

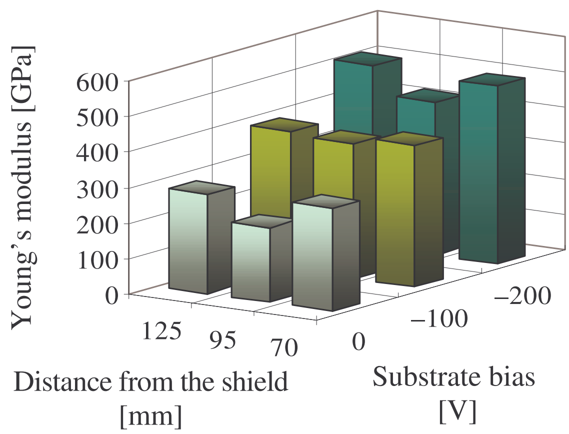

Figure 9. Effect of the substrate bias and distance of specimens from the magnetron disk on Young's modulus of the two-layer Ti+Ti(C,N) coatings.

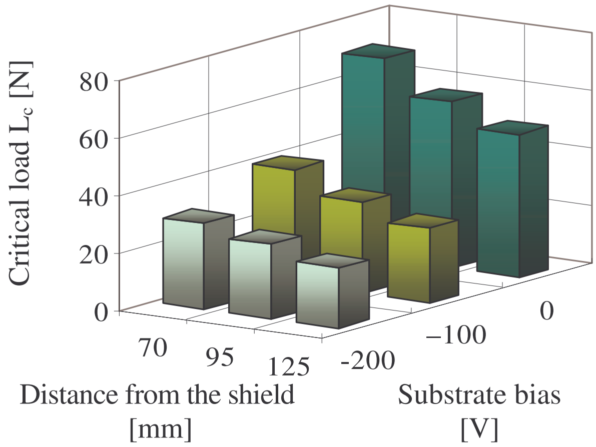

Figure 10. Effect of the substrate bias and distance of specimens from the magnetron disk on adhesion of the two-layer Ti+Ti(C,N) coatings to the substrate material.

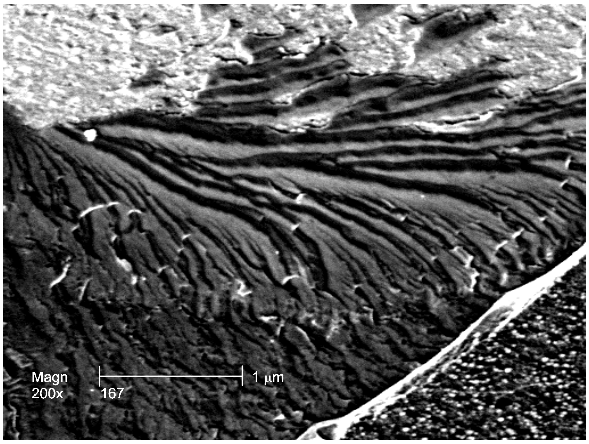

Figure 11. Defect of a Ti+Ti(C,N) coating (coating deposition conditions: substrate bias 0V, distance of specimens from the magnetron disk 95mm).

4. SUMMARY

It was found out, basing on the investigation carried out, that from among all of the developed Ti+Ti(C,N) coatings, the ones obtained at the substrate bias of 0V and distance from the magnetron disk of 70mm revealed the largest thickness of 11,38µm, whereas the smallest thickness of 3,4µm was revealed for coatings obtained at the substrate bias of –100V and specimens distance from the magnetron disk of 125mm. Specimens obtained at the substrate bias of –200V and at the distance of 125 mm from the magnetron disk reveal the highest micro-hardness of $3640HV_{0,05}$. Increase of N concentration in the analysed coatings along with the decrease of the substrate bias result in their hardness increase. Adhesion of the analysed coatings to the substrate decreases with lowering the substrate bias, which is accompanied by hardness increase.

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