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Properties determination of two-layer coatings deposited by PVD techniques using computer simulation

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ABSTRACT

Purpose: The aim of the research is the computer simulation of the internal stresses in bilayer coatings Ti+TiN and Ti+Ti(C,N) obtained in the magnetron PVD process on the sintered high-speed steel of PM HS6-5-3-8 in working atmosphere including 100% N_2, and 50%$N_2$50%CH_4.

Design/methodology/approach: The experimental values of stresses were determined basing on the X-ray diffraction patterns using method $\sin^2\Psi$ and computer simulation of stresses was carried out in MARC environment, with the help of finite elements method.

Findings: The computer simulation results correlate with the experimental results. The presented model meets the initial criteria, which gives ground to the assumption about its usability for determining the stresses in coatings, employing the finite element method using the MARC program.

Research limitations/implications: It was confirmed that using of finite element method for estimating stresses in PVD coatings can be a way for reducing the investigation costs Results reached in this way are satisfying and in slight degree differ from results reached by experimental method. However for achieving better calculation accuracy in further researches it should be developed given model which was presented in this paper.

Originality/value: Presently the computer simulation is very popular, what allows to better understand the interdependence between parameters of process and choosing optimal solution. The possibility of application faster and faster calculation machines and coming into being many software make possible the creation of more precise models and more adequate ones to reality

Keywords: Numerical techniques; Stresses; Computer simulation; Finite element method

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METHODOLOGY OF RESEARCH, ANALYSIS AND MODELLING

1. Introduction

Quick development of different industry parts nowadays determines more requirements concerning engineering materials in the range of mechanical properties, wear resistance, erosion influence and high temperature influence.

It is essential to understand phenomena proceeding during production, treatment and service of the engineering material so that it meets the requirements. The internal stress phenomenon, resulting from irregular heating or cooling, during plastic working treatment in the consequence of phase process at mechanical surface treatment, pouring and metal and alloy solidification and also during anti-wear coating deposition has also its impact. One has to perform various tests to acquire that, from destructive methods to the non-destructive ones, like the ultrasonic - and diffraction methods [2,3,4].

The advanced surface treatment technology, especially among others PVD technology, allows to improve service properties of tools made from the conventional tool materials, which - if not subjected to such treatment - in many cases do not meet the requirements and expectations of tools users. Specifications of the PVD coatings fabrication process require performing analysis of the process parameters effect on material the substrate and also interaction between the coating and material substrate in the intermediate zone[1, 5-8].

The finite element method is commonly used currently in such branches of science, like: mechanics, biomechanics, mechatronics, materials engineering, and thermodynamics. All types of simulations shorten the design process and give the possibility to investigate the particular factors on the entire model. This is often impossible to achieve in real conditions or not justified economically. The finite element method makes it possible to understand better the relationships among various parameters and makes it possible to select the optimum solution [9-16].

Many analyses are performed nowadays using advanced calculation techniques, among others the finite element methods (FEM). These methods allow to perform complex analyses of phenomena occurring during coating deposition processes. It should be mentioned that such analyses require knowledge of many quantities, which are analysis parameters and these parameters are, among others, the physical and mechanical properties of substrate and coating material and also parameters of the coating deposition process. Therefore, the FEM program allows to create the model that describes mechanical and service properties, which depends on process parameters and also on parameters of the materials of substrate and coating [17-20].

The paper presents a model enabling the user to evaluate the overall stresses in the examined test pieces and to evaluate the computer simulation results of the deposition conditions effect on stresses on the Ti+TiN and Ti+Ti(C,N) PVD coatings. The comparative analysis was carried out of the results of computer simulation of stresses with the experimental results.

2. Investigation methodology

Experiments were performed on the PM HS6-5-3-8 sintered high-speed steel test pieces, which were heat treated in a salt bath furnace with austenitizing at 1180° C and threefold tempering at 540°C. The Ti+TiN and Ti+Ti(C,N) coatings were put down onto the surface of the steel test pieces in a magnetron PVD process, at 460, 500 and 540°C. Structure of generated coatings was observed at lateral fractures on a High Resolution Scanning Electron Microscope SUPRA 35, ZEISS [7].

The chemical compositions of the coatings were determined using the glow discharge optical emission spectrometer GDOS.

X-rays studies for the analyzed coatings were carried out on X´Pert PRO system made by Panalytical Company using the filtered radiation of a cobalt anode lamp. The phase analysis of the analysed materials was carried out in Bragg-Brentano geometry using a Xcelerator strip detector.

In order to estimate the privileged growth direction of analyzed coatings, analysis of texture was performed. Not less than three pole figures were measured for each analysed sample made by a reflection method employing Euler's circle of diameter 187 mm in a range of the test pieces inclination angle from zero to 75˚[21].

Measurements of stresses for the analyzed coatings were made with the sin2 Ψ technique based on the X'Pert Stress Plus software, which contains, in a form of a necessary database, values of material constants. In the sin2Ψ method based on diffraction lines displacement effect for different Ψ angles, appearing in the conditions of stress of materials with crystalline structure, a silicon strip detector was used at the side of diffracted beam. Samples inclination angle Ψ towards the primary beam was changed in the range of 0°-75°[21].

The micro hardness tests of the coatings were carried out on the SHIMADZU DUH 202 ultra-microhardness tester. Young's modulus was calculated using the HARDNESS 4.2 program being a part of the ultra-microhardness tester system. Examinations of the coating thickness were made using the "kalotest" method, consisting the measurement of the characteristic parameters of the crater developed as a result of wear on the specimen surface caused by the steel ball with the diameter of 20 mm [2,6,7].

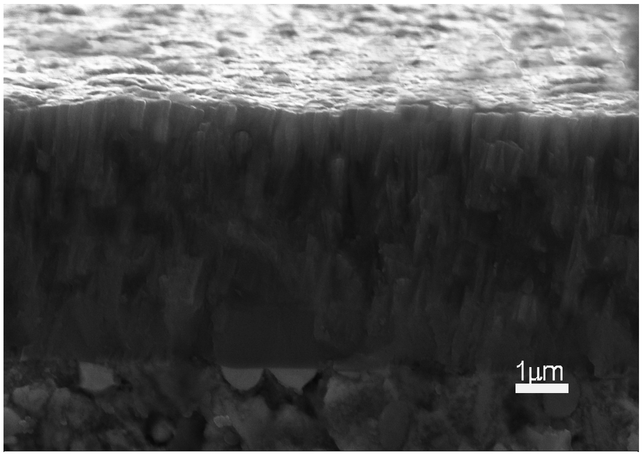


Fig. 1. Brittle fracture of a coating Ti+Ti(C,N) obtained in the magnetron PVD process, (the process temperature 500ºC)

Assessments for coatings adhesion to the substrate's material were made by a drawing method on Revetest device manufactured by CSEM.

The real specimen's dimensions were used for development of its model needed for determining the stresses in the coatings. The finite elements were used in computer simulation, basing on the 2D plane description, taking into account their central symmetry. The flat, axially symmetric PLANE 42 elements described by displacement in the nodes were used in simulation for the substrate, interface and the outer layer materials [9].

The geometrical model of tested coating with an applied mesh of finite elements. Conditions of spreading in those samples and their mechanical properties, which were determined in experimental way and used in computer simulation.

In order to carry out the simulation of internal stresses in Ti+TiN and Ti+Ti(C,N) coatings, the following boundary conditions were applied:

- symmetry axis of sample is fixed on the whole length by taking away the all degrees of freedom from nodes which are on this axis,
- change of temperature in PVD process presents the cooling process of specimen 460, 500 and 560°C to ambient temperature of 20°C,
- for TiN, Ti(C,N) coating an interface Ti and a substrate (PM HS6-5-3-8) materials properties were established on the basis of and Mat Web catalogue.

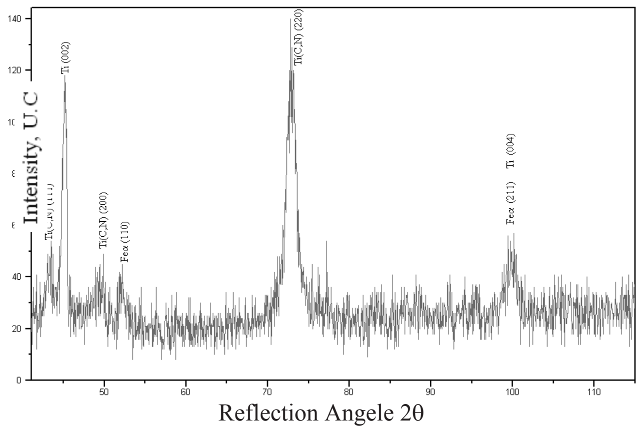


Fig. 2. X-rays diffraction patterns of the sintered high-speed steel PM HS6-5-3-8 with the Ti+Ti(C,N) coatings, (the process temperature 500ºC). Bragg-Brentano geometry

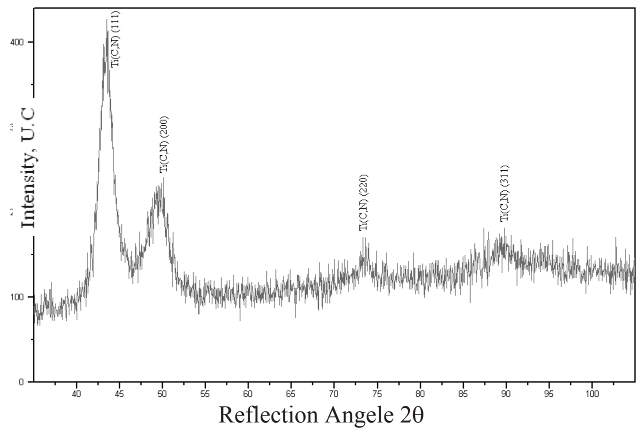


Fig. 3. X-rays diffraction patterns of the sintered high-speed steel PM HS6-5-3-8 with the Ti+Ti(C,N) coatings, (the process temperature 500ºC). Geometry of constant angle of incidence

3. Results

As a result of the factographic investigations performed on a scanning electron microscope, it was found that the deposited coatings determine column structure and particular layers are uniformly deposited and tightly adhere to each other as well as to the substrate's material (Fig. 1).

The chemical composition investigations for coatings made by a glow-discharge optical emission spectroscopy method, confirm Titanium, Nitrogen and Aluminium occurrence in the investigated coatings (Tables 2, 3).

The results of X-ray qualitative phase analysis confirmed occurrence of adequate phases in tested substrate and in coating material. (Figs. 2, 3). On the basis of performed pole figures (Figs. 4, 5) it was found that apart from temperature process of analyzed coating shows privileged <110> increase direction.

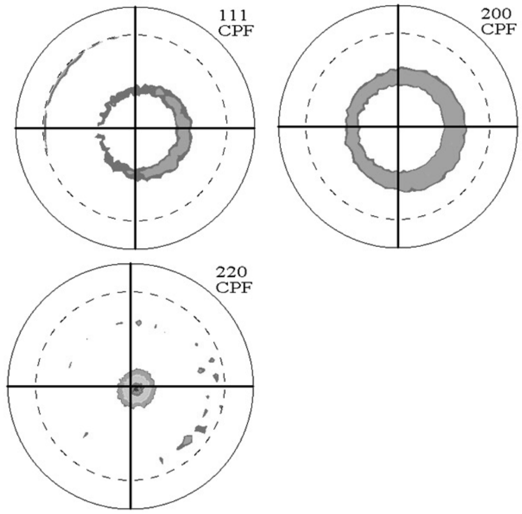


Fig. 4. Experimantal pole figures (111), (200) and (220) of Ti+Ti(C,N) coating, (the process temperature 500ºC)

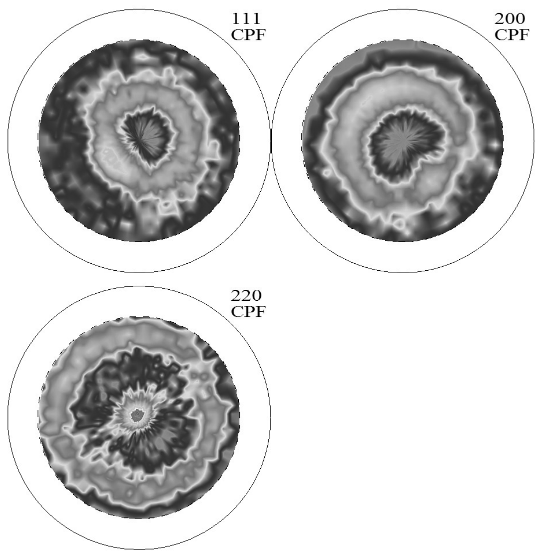
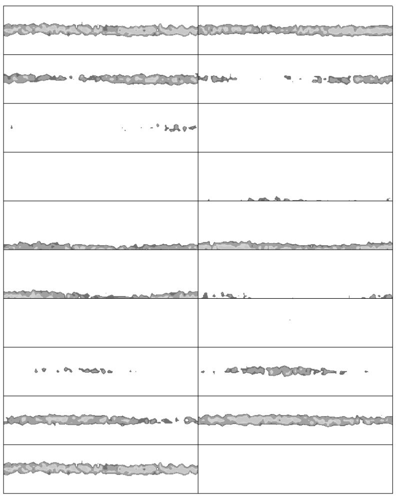


Fig. 5. Experimantal pole figures (111), (200) and (220) of Ti+TiN coating, (the process temperature 500ºC)

a)



b)

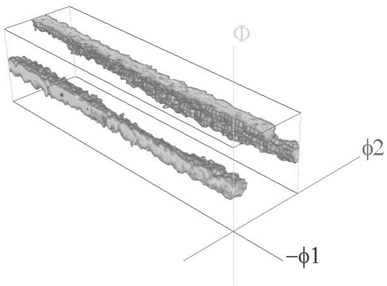


Fig. 6. A distribution function of coating orientation Ti(C,N) obtained in the magnetron PVD process on a substrate made of sintered high-speed steel PM HS6-5-3-8 in 540ºC; a) section acc. to φ_2 (for the following values φ_2: 0, 5, 10…90º), b) 3D FRO view after transformation

The stresses' assessment by $\sin^2\psi$ (Figs. 7 ,8) method is made based on analysis of reflexes (311) for the sake of the privileged <110> direction of their increase (Table.1).
Above-mentioned coatings are characterized by very good adhesion to substrate material what is indicated by high values L_c obtained during the time of parameter's measurement by "scratch- test" method. The results of carried out researches concerning internal stresses of analyzed coatings indicates the interdependence between stresses' value and their adhesion to substrate material.

Texture analysis for investigated coatings is carried out by a reflexion method. Concentric distribution of pole figures intensity changing along with a beam of those figures indicates occurrence of axial component texture of coatings acquired in the magnetron PVD process. Areas of intensity increase on recorded figures, independently on conditions to obtain coatings in the magnetron PVD process correspond to fibres presence <110> with direction close to a perpendicular direction in respect of sample plane. Depending on parameters needed to obtain a sample, fibres direction <110> is deviated from normal one against sample surface from 1 to 4°, thus, the distinguished axis deviation is dependent on vacuum furnace atmosphere and process temperature, and it is decreased when increasing supply current voltage in the course of a process of their depositing. Texture image of an exemplary coating acquired in the magnetron PVD process is presented in the form of experimental pole figures determined as CPF, complete pole figures calculated from FRO determined as RPF, pole figures determined from FRO (Fig. 6).

Using experimental and table data, internal stresses in coatings in environment MARC were modeled by the use of the finite elements method. Figs. 9, 10 presents obtained results of numerical analyze shown as stresses deposition maps in coatings Ti+TiN and Ti+Ti(C,N)). Numerical analyze showed existence of compress stresses in analyzed coatings, which don't exceed -1400 MPa. Tables 4, 5 presents results of stresses experimental determined and by the use of computer simulation and also results of thickness, adhesion and microhardness.

Table 1.
Texture volume fractions, deviation of distinguished axis and conditions to acquire coatings in the magnetron PVD process on a substrate made of sintered high-speed steel

Coatings	Process temperature, [°C]	Vacuum furnace atmosphere	Volume fraction for <110> component in areas covered by diffraction [%]
Ti+TiN	460°	100% N_2	59
	500°	100% N_2	53
	540°	100% N_2	46
Ti+Ti(C,N)	460°	50%$N_2$50%CH_4	53
	500°	50%$N_2$50%CH_4	35
	540°	50%$N_2$50%CH_4	55

Table 2.
The analyses of chemical composition of TiN coatings at sintered high-speed steel PM HS6-5-3

Process temperature,[°C]	Atomic concentration of the elements, [%]		
	Ti	N	Al
460^0	48	42	9
500^0	44	51	5
540^0	43	51	3

Table 3.
The analyses of chemical composition of Ti(C,N) coatings at sintered high-speed steel PM HS6-5-3

Process temperature, [°C]	Atomic concentration of the elements, [%]			
	Ti	C	N	Al
460	59	9	23	7
500	67	9	15	8
540	63	9	18	8

Table 4.
Results mechanical properties of TiN coatings at sintered high-speed steel PM HS6-5-3

Process temperature, [°C]	Thickness, [μm]	Microhardness [HV0,07]	Adhesion, [N]	Experimental stress results, [MPa]	Computer simulation stress results, [MPa]
460	6.8	2790	104	-860	-840
500	4.1	2620	87	-818	-850
540	3.8	2410	72	-792	-760

Table 5.
Results mechanical properties of Ti(C,N) coatings at sintered high-speed steel PM HS6-5-3

Process temperature, [°C]	Thickness, [μm]	Microhardness [HV0.07]	Adhesion, [N]	Experimental stress results, [MPa]	Computer simulation stress results, [MPa]
460°	4.6	2650	85	-1400	-1430
500	4.2	2590	35	-1215	-1200
540	3.9	2420	55	-1042	-1013

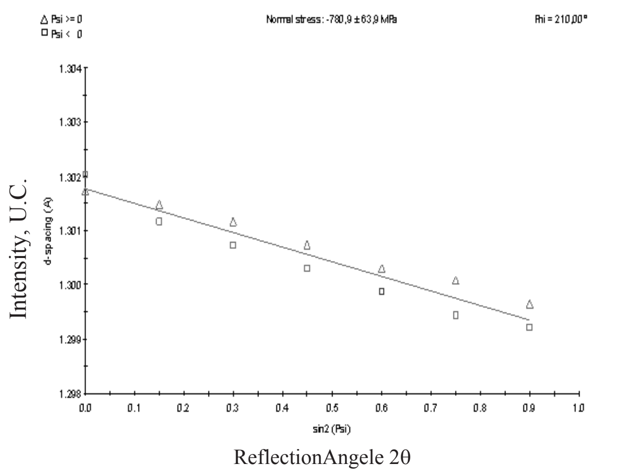


Fig. 7. Changes of interplanar distance of d reflex (311) of intermediate TiN layer as a function of $\sin^2\Psi$ stresses' measurement made by $\sin^2\Psi$method for different φ values of samples setting towards goniometer axis, Ti+TiN coating obtained on a substrate made of sintered high-speed steel PM HS6-5-3)

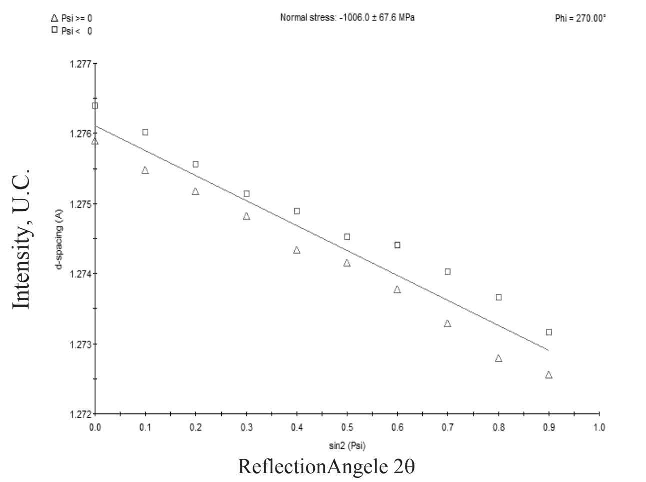
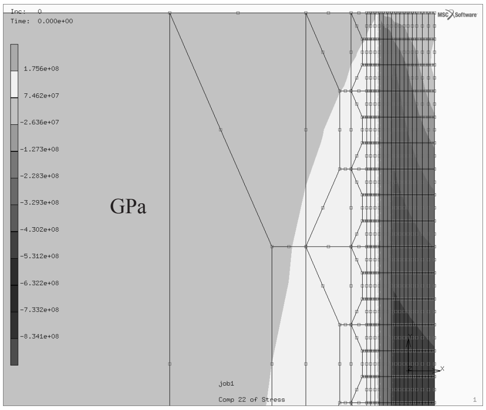


Fig. 8. Changes of interplanar distance of d reflex (311) of intermediate Ti(C,N) layer as a function of $\sin^2\Psi$ stresses' measurement made by $\sin^2\Psi$method for different φ values of samples setting towards goniometer axis, Ti+Ti(C,N) coating obtained on a substrate made of sintered high-speed steel PM HS6-5-3)

a)



b)

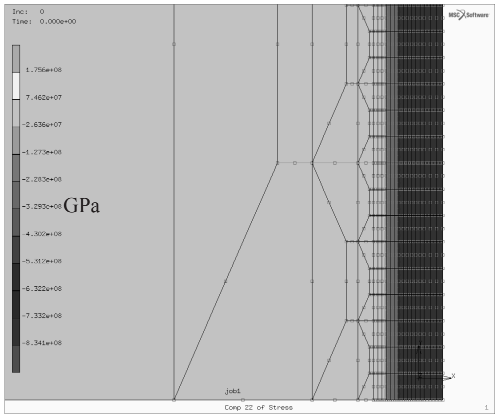
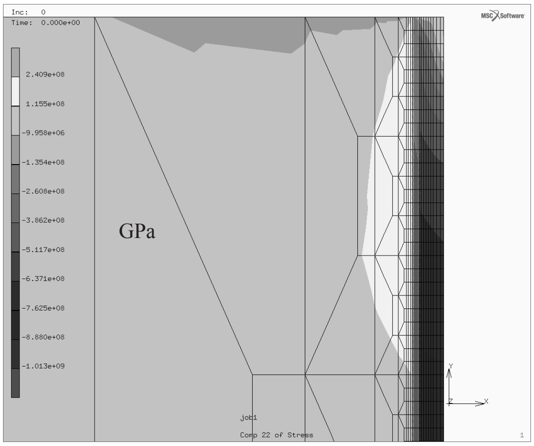


Fig. 9. Distribution of the simulated compression stresses in the TiN coating (coating thickness g=6.8 μm, process temperature 460°C a) Stress distribution of the edge, b) Stress distribution of the center

a)



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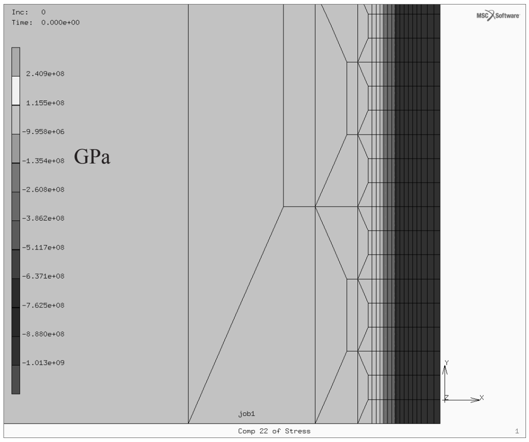

Fig. 10. Distribution of the simulated compression stresses in the Ti(C,N) coating, (coating thickness g=3.9 μm, process temperature 540°C, a) Stress distribution of the edge, b) Stress distribution of the center

4. Conclusions

In the result of analyzed texture it was found that tested coasting shows privileged <110> increase direction apart from conditions of their obtaining.

On the basis of performed researches it was found that the coatings shows higher values of compression stresses and shows better adhesion to substrate material and also higher microhardness.

The model presented in the project was developed by the use of the finite element method, using MARC program what makes possible to estimate internal stresses occurring in coatings Ti+TiN and Ti+Ti(C,N) obtained in magnetron process PVD. On the basis of the data concerning properties of material's substrate and coatings (Young's modulus, Poisson ratio, Thermal expansion coefficient) it is possible to estimate internal stresses in tested samples. The results of computer simulation correlate with results obtained in experimantal way.

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