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INTERNAL STRESSES IN PVD COATED TOOL COMPOSITES

The aim of work is the investigation of the internal stresses in PVD coated metal matrix composites (MMC). Sintered MMC substrate is composed of the matrix with the chemical composition corresponding to the high-speed steel, reinforced with the TiC type hard carbide phase. Functionally graded composition of MMC providing of high ductility characteristic of steel in the core zone as well as high hardness characteristic of cemented carbides in the surface zone. Internal stresses were determined with use of finite element method in ANSYS environment. The reason of undertaking the work is necessity of develop the research of internal stresses, occurring in the coating, as well as in the adhesion zone of coating and substrate, which makes it possible to draw valuable conclusions concerning engineering process of the advisable structure and chemical composition of coatings. The investigations were carried out on cutting tool's models containing defined zones differing in chemical composition.

Modelled materials were characteristic of chemical composition corresponding to the high-speed steel at the core, reinforced with the TiC type hard carbide phase with the growing fraction of these phases in the outward direction from the core to the surface, additionally coated with (Ti,Al)N or Ti(C,N) functionally graded PVD coatings.

Results of determined internal stresses were compared with the results calculated using experimental X-ray $\sin^2\psi$ method. It was demonstrated, that the presented model meets the initial criteria, which gives ground to the assumption about its utility for determining the stresses in coatings as well as in functionally graded sintered materials. The results of computer simulations correlate with the experimental results.

Keywords: Analysis and Modelling; Computational Materials Science; Finite Element Method; Stresses; Coatings PVD

1. Introduction

Presently the finite element method is one of the main methods of calculations in computer-aided engineering. In the most of the large and medium enterprises the beginning of product production is not able to start, before its estimates properties are positively verified using the FEM calculations [1-4].

In building advanced engineering systems, designers must go through a sophisticated process of modelling, simulation, visualization, analysis, designing, prototyping, testing, and lastly, production. It is worth to take into account, that much work is involved before the production of the final product. This is to ensure the workability of the final product and the profitability. Because of this, techniques which are connected with modelling and simulation in a fast and effective way have an influence on results in the application of the FEM that is repeated many times [5-8].

The finite element method makes it possible to understand the relationships among various factors better and makes it possible to select the optimum solution [9-12].

Very common use of machining processes makes it necessary to intensify research in area of modern technologies for deposition of avant-garde, wear-resistant coatings as well as proper substrate selection. Application of surface treatment technologies, as physical vapour deposition, has currently high development potential it area of methods for tool materials properties increase [13-14].

New generation of the metal matrix composite (MMC), functionally graded with the core sintered with the matrix of the chemical composition corresponding to the high-speed steel reinforced with the hard carbide (TiC) phase providing of high ductility characteristic of steel combined with high properties characteristic of cemented carbides. It can be achieved mainly because of the possibility of ensuring the change of the chemical composition by using growing fraction of hard phases in the outward direction from the core to the surface. It can have an influence on decrease of fabrication costs thanks to savings made on the hard carbide phase, used in the tool surface zone only. Additional reinforcing of the surface by PVD depositing of (Ti,Al)N or Ti(C,N) coating makes it possible to improve the efficiency of tools made from developed materials and to widen their range of application [15-16].

Internal stresses occurred in analyzed materials are the important material data as they have an important effect on structural phenomena in materials and their properties (hardness, cracking rate, fatigue resistance). Because of the functional properties of the coatings for the cutting tool flanks it is more advantageous that the coatings have the compression character of stresses, as heating the substrate up in the machining process should not lead to occur and develop

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of coating cracks, but only to reduction of the compression stress value, in the coating[17-21].

The general aim of this paper is the computer simulation with the use of finite element method (FEM) for determining the character and value of internal stress in PVD coated MMC materials and comparison of the obtained results with laboratory measurements. Sintered MMC substrate was composed of the matrix with the chemical composition corresponding to the high-speed steel, reinforced with the TiC type hard carbide phase. Functionally graded composition of MMC providing of high ductility characteristic of steel in the core zone as well as high hardness characteristic of cemented carbides in the surface zone. Such composed material was sintered, heat treated and PVD deposited with Ti(C,N) or (Ti,Al)N coatings.

2. Investigation methodology

The investigations were performed on the samples which were made with the help of unilateral uniaxial moulding, sintered in the pipe furnace in the protective atmosphere. The chemical composition of steel powder that was used as the matrix in tested sintered composite materials presents Table 1. The powder mixes were moulded in a die pouring it in 4 sequenced layers with the successive lots of mixes with the growing content of reinforcing phases, cut-and-fill balancing each time the surface of a layer about 0.7 mm thick. The thickness of the bottom layer, containing pure high-speed steel powder was about 1.9 mm. The aim of such composition was decreasing of distortion during the sintering process. The layer with the maximum fraction of reinforcing phases, as characterized by the least moulding capability, was poured as the last one to obtain the maximum pressure in the area just under the punch during the uniaxial moulding process. Structure observations, X-ray qualitative and quantitative microanalysis of the investigated materials were made with the use of ZEISS SUPRA 35 scanning electron microscope at the accelerating voltage of 20kV.

Adhesion evaluation of the coatings on the tested inserts was performed with the help the scratch test on the CSEM REVETEST device. This device moved the diamond penetrator along the examined samples' surface with the gradually growth of load. The tests were performed with the following parameters: load range 0-100 N, load growth rate (dL/dt) 100 N/min, penetrator's travel speed (dx/dt) 10 mm/min, acoustic emission detector's sensitivity AE 1. The critical load LC, at which coatings' adhesion is lost and was defined on the grounds of the registered values of the acoustic emission AE and friction coefficient Ft.

Microhardness inquiry for produced coatings and substrates hardness were carried out by means of with the help of ultramicrohardness tester - DUH 202 produced by Shimadzu Co. using Vickers method at load of 0.05 N for coatings acquired in PVD process and 0.07 N

Stresses distribution tests were done in ANSYS software using dimensioning model shown on Fig. 1. Analysis were carried out in two variants of materials. Individual variants differ in kind of used PVD coating (Table 2). In order to achieve assumed purposes of this paper, it was also assumed the simplified model of structured materials with zones division including established mechanical and physical properties. For all zones of individual variants, these properties were selected and put into the computer programme that makes the analysis such as Poisson ratio,Young's modulus, thermal expansion coefficient, density.

TABLE 1

Steel matrix powder: mass concentration of the elements,[%]

Steel matrix powder: mass concentration of the elements,[%]						
C	Mn	Si	Cr	W	Mo	V
0.84	0.36	0.35	3.97	6.54	4.81	1.95

Change of temperature in PVD process presents the cooling process of the specimen from 500 °C to ambient temperature of 20°C. Materials properties used for simulation input was presented in table 3.

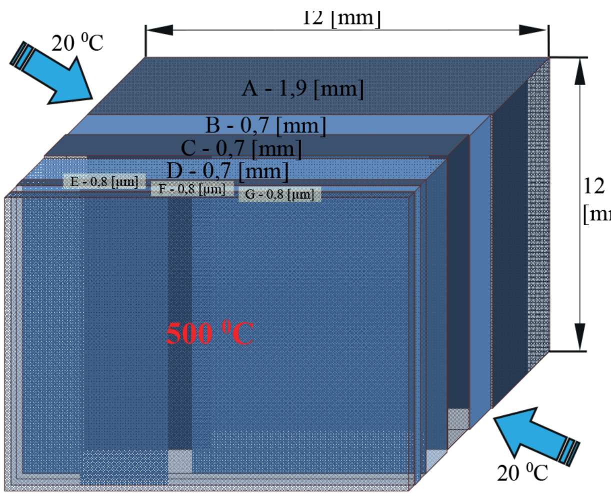

Fig. 1. Dimensioning model in SD section

It is necessary to take into account the fact that significant difference between the thickness of three top layers and these that are in the base of model, dimensions of each model were put in calculation programme using scale, what was presented in table 2. One millimetre in a real model responds to one micrometer of the model that was made with the help of ANSYS software.

X-rays studies in the analyzed materials were carried out on X´Pert PRO system made by Panalytical Company using filter radiation of a cobalt anode lamp. A phase analysis of the analysed materials were carried out in Bragg-Brentano geometry with X-rays quality using a Xcelerator strip detector. Measurements of stresses for the tested coatings were performed by sin2ψ and technique by X'Pert Stress Plus company's programme, which contains values of material constants, in a form of a database indispensable to calculate.

In the method of $\sin^2\psi$ (Fig. 2) based upon diffraction lines and displacement effect for various ψ angles, occurring in the conditions of the materials stress with the crystalline structure, a silicon strip detector was applied at the side of diffracted beam. Specimen inclination angle ψ towards the primary beam was varied in the range of 0° ÷ 75°.

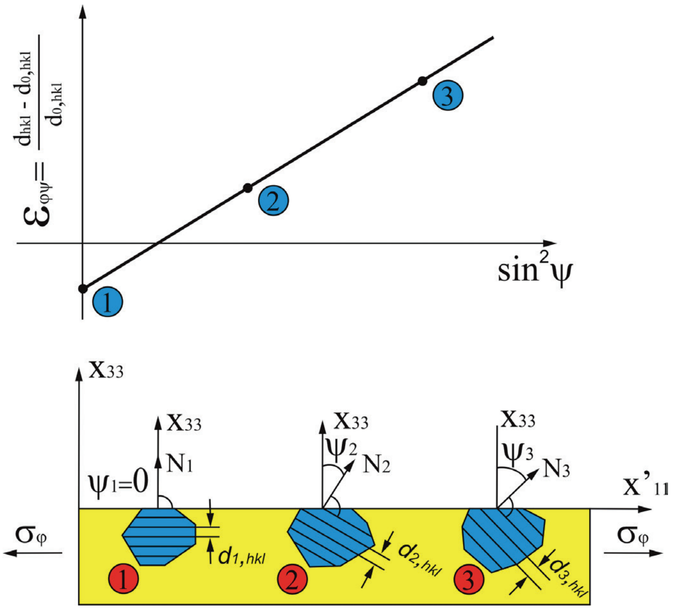

Fig. 2. Linear dependence in a classical method of $\sin^2\psi$ significant for assumptions of the homogenous and flat state of stresses; points 1, 2, 3 correspond to measurements of values for interplanar distance at proper - oriented grains of microstructure in different directions at ψ angle

The formulated model was experimentally verified by making the comparison of calculated results with experimental research. The procedure of verification consisted in evaluating of results significance in computer simulation of stresses relatively to experimental results obtained during stress measurement.

3. Results and discussion

It was found out, based on the structure observations of developed composite materials, that there were no fractures and delamination between poured in matrix and moulded layers. Microstructure of particular zones of investigated composite materials obtained during SEM observations were shown in Figs. 3-5, additionally in Fig. 3 the chemical composition of (Ti,Al)N coating deposited on sintered composite was introduced. The coating is characteristic of compact structure with good adhesion to zone 4 without any delamination. Sintered substrate (zones 1-4) are characteristic of dense, structure with locally occurred pores. There conglomerates of carbides at the grain boundary areas were observed. There was no releases of large-sized carbides found during the carried out observations (Figs 4-5).

High adhesion of the analyzed coatings, defined by load value Lc responsible for coating's destruction is adequately 76 & 82 N depending on investigated variants.

Phase and chemical composition, conditions and type of process as well as substrate's material and combination of the applied layers and texture influence on microhardness of the investigated coatings (2860 HV0.07 in case of Ti(C,N) coating and 3120 HV0.07 in case of (Ti,Al)N coating).

TABLE 2

Configuration of individual zones in models

	VARIANT 1 functionally graded MMC with Ti(C,N) coating	VARIANT 2 functionally graded MMC with (Ti,Al)N coating
Zone 7	TiC	(0.3 Ti, 0.7 Al)N
Zone 6	Ti(C,N)	(0.3 Ti, 0.7 Al)N
Zone 5	TiN	TiN
Zone 4	Steel matrix + 11%TiC	
Zone 3	Steel matrix + 7%TiC	
Zone 2	Steel matrix + 3%TiC	
Zone 1	Steel matrix	

TABLE 3

Properties of individual layers

Material	Young's modulus.	Poisson ratio	Thermal expansion coefficient,
	*10^11 [Pa]	[-]	*10^-6 [K^-1]
	EX	NUXY	ALPX
Steel matrix	0.215	0.30	11.5
Steel matrix +3%TiC	0.225	0.30	11.2
Steel matrix +7%TiC	0.233	0.30	10.7
Steel matrix +11%TiC	0.241	0.29	10.1
TiN	0.510	0.26	9.5
(Ti,C)N	0.410	0.24	9.4
TiC	0.390	0.19	7.8
(0,7%Ti,0,3%Al)N	0.420	0.23	7.9
(0,3%Ti,07%Al)N	0.480	0.27	8.3

The results are the base for a statement, that material that serves for experimental verification of modelling results is compatible with an assumed model. High structure compatibility and comparative material properties with a model that was tested in simulation which gives the possibility of obtaining comparative results of experimental researches with high precision relating to modelling results.

Employed methods of obtaining the proper phase composition (being a consequence of chemical composition), the adhesion of analyzed coatings to applied substrate's material correlating with a value of internal stresses, the applied combination of layers, and also a shape of surface topography decided about obtained mechanical and functional properties.

The internal stresses were modelled on the basis of experimental results and assumpted datas (shown in Table 3), using ANSYS software, with the finite element method. Table 5 presents calculated results of stresses range value in individual zones for all analyzed variants of material while Figures 5-6 present graphical comparison obtained results of modelling.

The maximum stresses value ascends while changing of analyzed zone towards the surface of the sample (zone 5-7) and reach the level over (-)1070 MPa in zone 7, in area near to edges. Concentration of compressive stresses in edges might has influenced on higher microhardness of tested material and also contributing to improvement of its functional properties (Figs 6-7).

Calculated values of internal stresses in tested metal matrix composites deposited with PVD coatings change gradually towards surface of coatings. Thanks to precise composition of functionally graded substrates and proper selection of coatings properties, it was achieved small difference of stresses between zone 4, which is real zone of substrate surface for PVD coating, and zone 5 which is first coating zone (Figs 6-7). It contributes to more coating adhesion to substrate of material and benefits from functional properties of tested materials (Table 4).

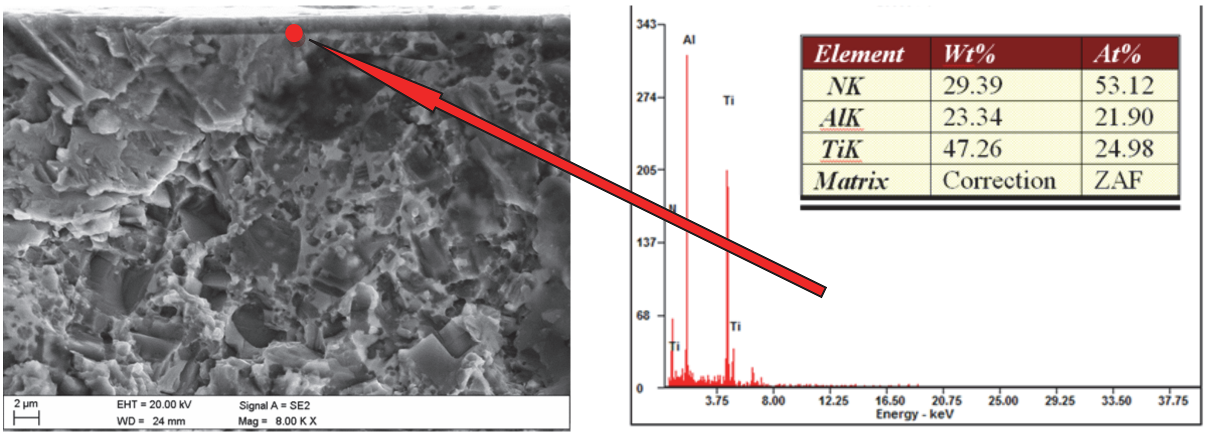

Fig. 3. Microstructure of zone 4 of sintered substrate and zones 5-7 of (Ti,Al)N coating

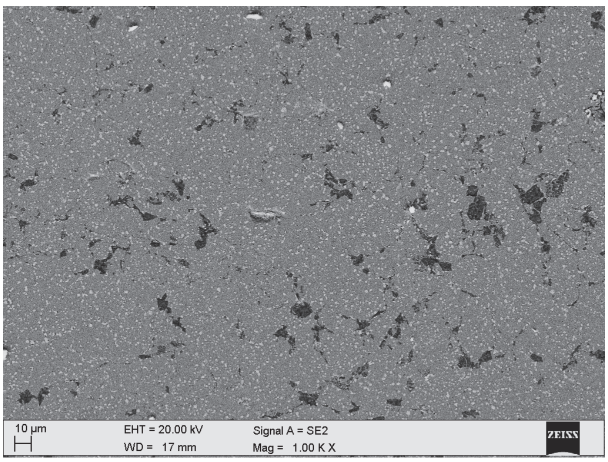

Fig. 4. Microstructure of zone 2

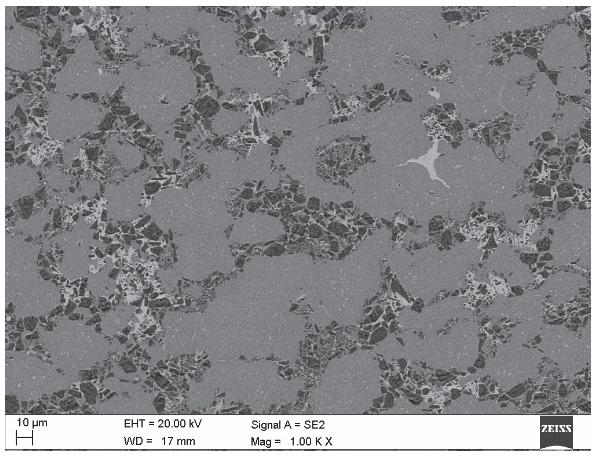

Fig.5. Microstructure of zone 4

TABLE 4

Comparison of adhesion and microhardness of deposited coatings

	VARIANT 1 functionally graded MMC with Ti(C,N) coating	VARIANT 2 functionally graded MMC with (Ti,Al)N coating
Critical load Lc, N	76	82
Microhardness $HV_{0.07}$	2860	3120

TABLE 5

Computer simulation results of internal stresses for all variants in particular zones [MPa]

	VARIANT 1 functionally graded MMC with Ti(C,N) coating	VARIANT 2 functionally graded MMC with (Ti,Al)N coating
Zone 1 (1.9mm)	(-) 0-151	(-) 0-150
Zone 2 (0.7mm)	(-) 0-160	(-) 0-161
Zone 3 (0.7mm)	(-) 0-114	(-) 0-113
Zone 4 (0.7mm)	(-) 0-120	(-) 0-117
Zone 5 (0.8μm)	(-) 263-416	(-) 258-415
Zone 6 (0.8μm)	(-) 251-371	(-) 447-553
Zone 7 (0.8μm)	(-) 874-981	(-) 915-1070
Results of experimental investigations of stresses, sin2ψ method		
Zone 7 (0.8μm)	(-) 1002 ± 45.8	(-)1006 ± 67,6

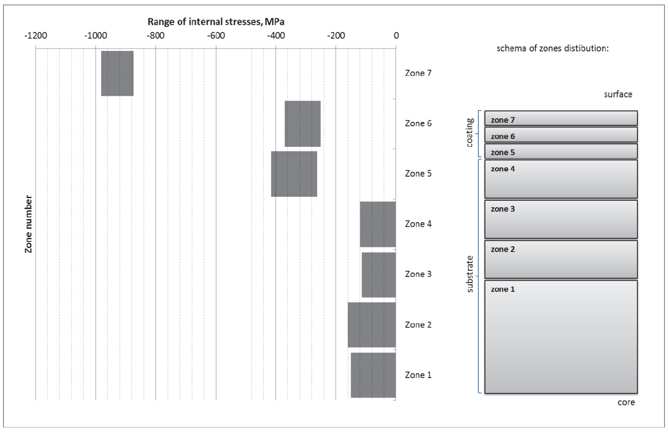

Fig. 6. Graphical comparison of range of internal stresses in particular zones simulated for functionally graded MMC deposited with Ti(C,N) coating

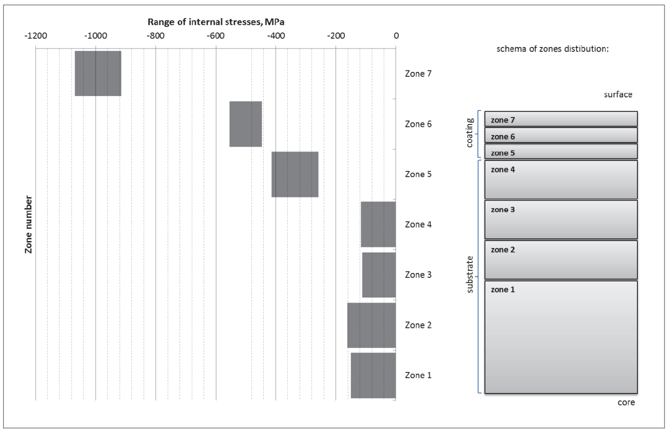

Fig. 7. Graphical comparison of range of internal stresses in particular zones simulated for functionally graded MMC deposited with (Ti,Al)N coating

The results of carried out simulations were verified by carrying out the laboratory research in the range of average values of internal stresses in deposited coatings. Figure 9 presents change of interplanar distance d for (311) reflection of (Ti,Al)N coating in function of $\sin^2\psi$.

Metal science laboratory research, in many cases, only allows for the measurement of selected quantities and parameters in limited fields in respect to the complex form and value variables at the intersections of examined elements, so experimental laboratory research can be fulfilled only in case of outer zone of investigated material (Zone 7).

In case of investigated PVD coatings, the stresses' assessment by $\sin^2\psi$ method was carried out based on (311) reflexes analysis (Fig. 8) for the sake of the privileged <110> direction of their increase. For coatings acquired in the arc PVD process, stresses are defined on the ground of analysis of reflex displacement at the highest value of 2θ angle. Analysed reflexes were free from influencing on its shape and location of the other components of the investigated material (substrate's material, possible other layers included in the coating). Locations of the reflexes were determined with use of a Gaussian curves matching method. In the method of sin2ψ, the reflexes recorded at higher values of the 2θ angle are preferred considering higher sensitivity of deformations and lower error of obtained results. Nevertheless, is not always possible to achieve that in experimental studies, when not enaugh peaks intensity, resulting from extensive or irregular shape and texture of analyzed coatings, as well as relatively small depth of penetration of X-rays radiation high angles range of reflexion make correct and proper assessment of theirs impossible.

In order to confirm the reliability of results it was done the verification of properly formulated statistic hypothesis concerning the results compatibility of simulation and the measurements, admitting the level of significance $\alpha = 0.05$.

Basing on test results with small amount it was used test t-Student's for average value population.

It was analyzed the following hypothesis: High compatibility between the measurement results of obtained stresses in measuring method and the results obtained in stresses simulation. In this case it was accepted the null hypothes in the form:

$$H0 : \mu = \mu 0$$

where $\mu 0$ – is the value of stresses obtained in the result of computer simulation, and hypothes was verified basing on measurement test of stresses which was obtained by the experimental method.

It was stated the compatibility of computer simulation results with the result concerning the stresses obtained by the use of experimental method for assumed of significance level (Table 6). The results of verification present unequivocally correctness of model and its full adequacy.

TABLE 6

Statement of results from significance test of correlation coefficient for computer simulation and experimental results of stresses

Correlation coefficient r Pearsona	Value of t-Student's test	The level of significance for test t	Result of test
0.653567	5.662440	0.000001	Significant

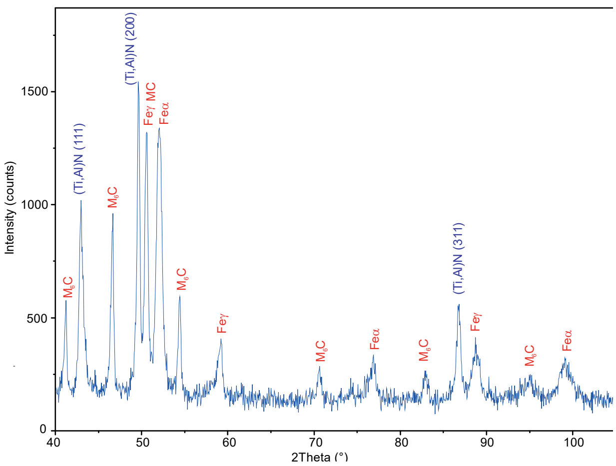

Fig. 8. Diffraction patterns of the steel matrix +(Ti,Al)N variant of PVD coated high-speed steel matrix composite

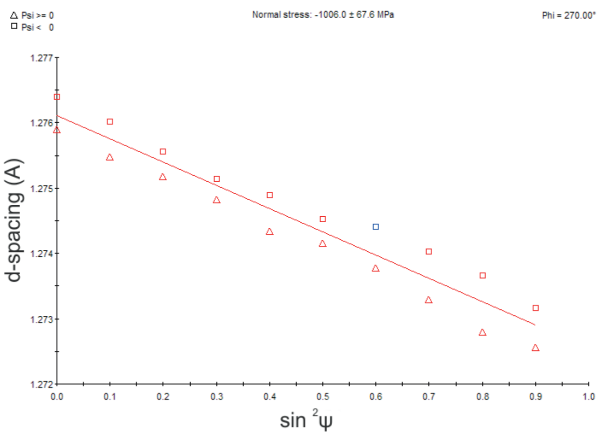

Fig. 9. Change of interplanar distance d for (311) reflection of (Ti,Al) N coating in function of sin $^2\psi$

4. Conclusion

Many papers [8-11] are dedicated to process' parameters influence of coatings' deposition, substrates' polarisation and temperature, reactive gases pressure and flow on their properties expressed through adhesion, hardness, internal stress status, phase composition and crystallographic orientation, and also a type of structure. Knowledge status on dependencies among structure, physical properties and conditions to acquire coatings in the PVD & CVD processes is still unsatisfactory and it requires laboratory investigations, supported by computer technology. Applying the computer technology significantly increases analysing possibilities of received experimental results and reduces necessity to run costly and time-consuming technological tests in favour of mechanical and service properties prediction of coatings obtained in the PVD & CVD processes on tool materials.

Coating surface, directly endangered to a contact with foreign material, should be characterized by low chemical reactivity. The middle part of the coating section is required to be of high hardness and good ductility providing a possibility for internal stress relaxations. Zone of coating contact with material substrate should provide, above all, good adhesion, which can be obtained by thermal stresses minimization as well as similar character of bonds between atoms both in coating and substrate.

In every verified case occurred compress stresses of little or medium values which could have positive influence on functional properties of analysed materials. It was also noticed that maximum value of stresses gradually ascends towards the surface of sample and gets the maximum value in zone close to edges. This gathering of compress stresses in edges could has influence on higher microhardness of tested material and benefiting also from improvement of its functional properties. It was able to get minimum difference of stresses among surface of substrate and PVD coating with the help of applied structure of substrates and coatings. It benefits from good coating adhesion with substrate of material and positively influence on functional properties of analysed materials.

Results of determined internal stresses were compared with the experimental results of stresses calculated using the X-ray $\sin^2\psi$ method. It was demonstrated, that the presented model meets the initial criteria, which gives ground to the assumption about its utility for determining the stresses in coatings as well as in functionally graded sintered materials.

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REFERENCES

[1] E. Vogli, W. Tillmann, U. Selvadurai-Lassl, G. Fischer, J. Herper, Appl. Surf. Sci. **257**, (20), 8550–8557 (2011).

[2] Q. Wang, F. Zhou, X. Wang, K. Chen, M. Wang, T. Qian , Y. Li, Appl. Surf. Sci. **257**, (17), 7813–7820 (2011).

[3] M. Marvi-Mashhadi, M. Mazinani, A. Rezaee-Bazzaz, Comp. Mater. Sci. **65**, 197-202 (2012).

[4] F.R. Liu, Q. Zhang, W.P. Zhou, J.J. Zhao, J.M Chen. J. Mater. Process. Tech. **212**, (10), 2058-2065 (2012).

[5] A. Sliwa, M. Bonek, J. Mikuła. Appl. Surf. Sci. **18**, 1-6, (2016).

[6] L.A. Dobrzanski, M. Staszuk, K. Gołombek, A. Śliwa, M. Pancielejko, Arch. Metall. Mater. **55**, (1), 187-193 (2010).

[7] L.A. Dobrzański, A. Śliwa, W. Kwasny, J. Mater. Process. Tech. **164-165**, 1192-1196 (2005).

[8] L.W. Żukowska, A. Śliwa, J. Mikuła, M. Bonek, W. Kwaśny, M. Sroka, D. Pakuła D, Arch. Metall. Mater. **61**, (1), 149-152 (2016).

[9] A. Zieliński, G. Golański, M. Sroka, Kovove Mater. **54**, (1), 61-70 (2016).

[10] T. Tański, K. Labisz, K. Lukaszkowicz, A. Śliwa, K. Gołombek, Surf. Eng. **30**, (12), 927-932 (2014).

[11] A. Śliwa, J. Mikula, K. Golombek, T. Tanski, W. Kwasny, M. Bonek, Z. Brytan, Appl. Surf. Sci. **23**, 1-7, (2016).

[12] J. Montalvo-Urquizo, P. Bobrov, A. Schmidt, W. Wosniok, Mech. Mater. **47**, 1-10 (2012).

[13] A. Zieliński, G. Golański, M. Sroka, T. Tański, Mater. High Temp. **33**, (1), 24-32 (2016).

[14] Ł. Szparaga, J. Ratajski, Eng. Mat. **32**, 760-763 (2011).

[15] L.A. Dobrzański, W. Sitek, M. Krupiński, J. Dobrzański, J.Mater. Process. Tech. 157, 102-106 (2004).

[16] L.A. Dobrzański, D. Pakuła, Mater. Sci. Forum. **513**, 119-133 (2006).

[17] A. Zieliński, G. Golański, M. Sroka, P. Skupień, Mater. High Temp. **33**, (2), 154-163 (2016).

[18] L.A. Dobrzanski, W. Sitek, J. Mater. Process. Tech. **157**, 245-249 (2004).

[19] A. Zieliński, M. Miczka, B. Boryczko, M. Sroka, Arch. Civ. Mech. Eng. **4**, 813-824 (2016).

[20] A. Zieliński, G. Golański, M. Sroka, J. Dobrzański, Mater. Sci. Tech-Lond. (2016), DOI: 10.1179/1743284715Y.0000000137 (in press).

[21] Z. Shiping, E. Oubai, A. Rooh, A. Khurram , G. Wagdi Habashi, Finite Elem. Anal. Des. **57**, 55-66 (2012).