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THE EFFECT OF PVD AND CVD COATING STRUCTURES ON THE DURABILITY OF SINTERED CUTTING EDGES

WPŁYW STRUKTURY POWŁOK PVD I CVD NA TRWAŁOŚĆ SPIEKANYCH OSTRZY SKRAWAJĄCYCH

In the work it was demonstrated that the exploitative stability of edges from tool ceramics and sintered carbides coated with gradient and multilayer PVD and CVD coatings depends mainly on the adherence of the coatings to the substrate, while the change of coating microhardness from 2300 to 3500 HV0.05, the size of grains and their thickness affect the durability of the edges to a lesser extent. It was found that some coatings showed a fine-grained structure. The coatings which contained the AlN phase with hexagonal lattice showed a considerably higher adhesion to the substrate from sialon ceramics rather than the coatings containing the TiN phase. Better adherence of the coatings containing the AlN phase with hexagonal lattice is connected with the same kind of interatomic bonds (covalent) in material of both coating and ceramic substrate. In the paper the exploitative properties of the investigated coatings in the technological cutting trials were also determined. The models of artificial neural network, which demonstrate a relationships between the edge stability and coating properties such as: critical load, microhardness, thickness and size of grains were worked out.

Keywords: Tool Materials, PVD and CVD coatings, Surface Treatment, Machining, Artificial Neural Network

W pracy wykazano, że trwałość eksploatacyjna ostrzy skrawających z ceramiki narzędziowej i węglików spiekanych pokrytych gradientowymi i wielowarstwowymi powłokami PVD oraz CVD zależy głównie od przyczepności powłok do podłoża, natomiast zmiana mikrotwardości w zakresie od 2300 do 3500 HV0,05, wielkości ziarn oraz ich grubości w mniejszym stopniu wpływają na trwałość ostrzy. Powłoki wykazują drobnoziarnistą strukturę. Powłoki zawierające fazę AlN o sieci heksagonalnej wykazują lepszą przyczepność do sialonowego podłoża niż powłoki zawierające fazę TiN. Lepsza przyczepność powłok zawierających fazę AlN o sieci heksagonalnej związana jest z takim samym rodzajem wiązań międzyatomowych (kowalencyjnych) w materiale powłoki i ceramicznego podłoża. W pracy określono także własności eksploatacyjne powłok w technologicznej próbie toczenia. Zależności pomiędzy trwałością ostrza a własnościami powłok takimi jak obciążenie krytyczne, mikrotwardość, grubość i wielkość ziarna określono z zastosowaniem sztucznych sieci neuronowych.

1. Introduction

Numerous research studies have been devoted recently to coated tool materials, including also coated tool ceramics. It has been demonstrated that the designing of the coating-cutting edge system is based on such a selection of coating material which would reduce or totally eliminate the dominating wear mechanism of the cutting edge. As it has been demonstrated by numerous research studies, the coating should satisfy many requirements to ensure an appropriate protection of the tool during the machining. Literature studies show that the most important properties of coatings determining their exploitative advantages include undoubtedly microhardness, adhesion to substrate, thickness and grain size. Therefore, the paper has been focused on the research aiming to estimate the influence of coating properties of the coated cutting edges [1-11].

The application of systems supporting the design process is a necessity to be followed in our current economy. The increase of computing power observed over the last years contributes to the development of modern information technology tools applied to improve the quality of a product and to reduce its price. A special attention has been given to the systems developed for several years based on artificial intelligence algorithms and used to predict exploitative properties of the manufactured cutting edges on the basis of coating properties applied on sintered cutting edges. It can provide the manufacturers of cutting edges with the knowledge involving the exploitative durability of coated cutting edges without the necessity to repeat the expensive and long-lasting cutting ability trials. The capability to model the properties of materials is therefore extremely valuable for the manufacturers and designers of modern cutting tools since it means that the requirements of customers involving the quality of the delivered products can be satisfied. The modeling of coating properties is also connected with financial advantages since the expensive and time consuming research is reduced to a necessary minimum, that is to the research indispensable to

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verify the predicted quantities [12-15]. In spite of the fact that for many years intensive research studies have been carried out in various scientific centers on the elaboration of mathematical models enabling the determination of mechanical properties, they have not managed to work out a model which would comprehensively predict the exploitative properties of cutting tools coated with PVD or CVD.

The main objective of the present paper is to investigate the structure and properties of multilayer and gradient coatings obtained using the PVD and CVD techniques on sialon tool ceramics and sintered carbides and to define the influence of such properties of the coatings as microhardness, adhesion, thickness and grain size on the applicability properties of cutting edges covered by such coatings.

2. Materials

The research was carried out on multi-point inserts made of sialon tool ceramics and sintered carbides of WC-Co type, non-coated and coated with PVD and CVD. The inserts were coated in the cathode arc evaporation process CAE-PVD with the coatings of the type Ti(B,N), (Ti,Zr)N, Ti(C,N), Ti(C,N)+(Ti,Al)N, (Al,Ti)N, (Ti,Al)N and (Al,Cr)N, and in the high-temperature CVD process with multilayer coatings of the type Ti(C,N)+Al_2O_3+TiN and Ti(C,N)+TiN.

3. Methodology

The structure and morphology of the obtained coatings was observed in the scanning electron microscope. The observations of the structure of thin foils and the diffraction tests were carried out in the transmission electron microscope. The concentration changes of the chemical elements of the coating in the direction perpendicular to its surface and the concentration changes in the interphase zone between the coating and substrate material were determined basing on the tests in the glow discharge optical spectrometer. The analysis of phase composition of the substrates and of the obtained coatings was carried out with the use of X-ray diffraction method. The assessment of grain size in the investigated coatings was carried out using the XRD patterns obtained with the application of the grazing incident X-ray diffraction technique (GIXRD) using the Scherrer's method. The measurements of microhardness of the substrates were carried out using the classical Vickers method and the microhardness of the produced coatings was carried out using the dynamic method of Vickers in the mode 'load-unload'. The adhesion of the coatings to the substrate was determined basing on the Scratch Test analysis. The critical load L_c at which the adhesion of the coating fails was determined basing on the value of acoustic emission recorded during the measurement and on the observation of scratches formed during the scratch test. Detailed observations of the formed damage were carried out on the scanning electron microscope. In order to categorize the investigated machining inserts according to their usability properties, technological cutting trials were carried out. The durability of the investigated inserts was determined basing on the measurements of the width of wear band on the tool flank. The machining trials were being stopped when the assumed wear criterion for after-machining of VB=0.2 mm was exceeded.

Basing on the set of experimental results, a model of artificial neural networks (SSN) was elaborated, which made it possible to determine if there is a dependency between the properties of the coatings such as microhardness, adhesion to substrate, grain size or coating thickness and the durability of cutting edges covered with the investigated coatings. We investigated the possibility to apply networks of different architecture, and from among the tested networks the best quality factors were obtained for the network of multilayer perceptron (MLP) with one hidden layer. The network was trained out using the error backpropagation algorithms and conjugate gradients. To verify the usefulness of the network we applied the average absolute error, standard deviation quotient and Pearson's correlation factor.

4. Results and discussion

In the investigated coatings there occur secondary isomorphic solid solutions with titanium nitride TiN and also titanium carbonitride Ti(C,N), aluminium nitride AlN of the hexagonal lattice, chromium nitride CrN and aluminium oxide Al_2O_3 in the case of CVD coating. On the XRD patterns from the investigated coatings we also found the reflexes from the phases occurring in the substrate material, which is effected by the thickness of the obtained coatings, lower than the penetration depth of X-rays into the material (Fig. 1a). In effect of the research with the use of grazing incident X-ray diffraction (GIXRD) at low incidence angles of prime X-ray beam, we recorded only reflexes from thin surface layers (Fig. 1b,c). Basing on the obtained XRD patterns with the grazing incident X-ray diffraction, the models of coating structure were determined (Fig. 1d). The grain size of the investigated PVD coatings is within 8.2÷57 nm and it is lower than the grains of CVD coatings being within the range of 112÷421 nm (Table 1).

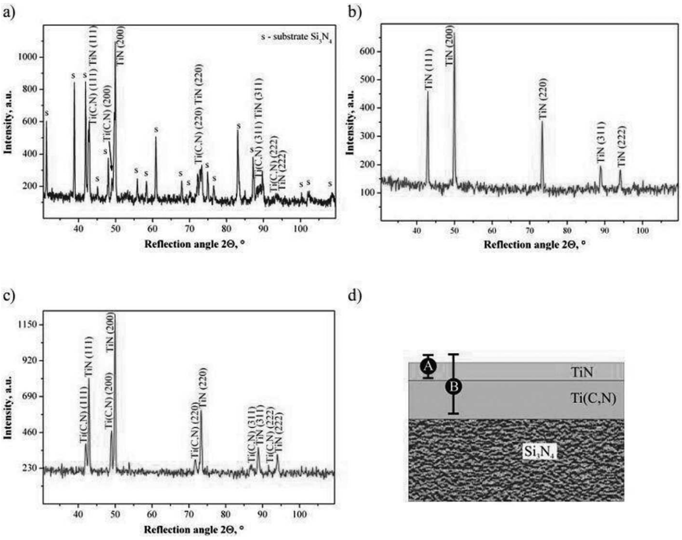

Fig. 1. X-ray diffraction pattern of Ti(C,N)+TiN coating deposited on the sialon ceramics obtained by a) Bragg-Brentano method, b, c) GIXRD method (α =0.5 and 3°), d) Scheme of packing layers into Ti(C,N)+TiN coating, which was deposited on sialon tool ceramic with marking depths of GIXRD phase analysis: A for α = 0.5°, B for α = 3°

TABLE 1

Mean values of thickness, microhardness, critical load, grain size and cutting ability of investigated samples

Substrate	**Coating**	**Thickness, μm**	**Micro-hardness, HV 0.05**	**Critical load Lc, N**	**Grain size, nm**	**Cutting ability T, min**
Sintered carbieds	uncoated	-	1826	-	-	2
	Ti(B,N)	1.8	2951	34	21	15
	(Ti,Zr)N	3.0	2842	40	21.4	13
	Ti(C,N) (1)	2.1	2871	49	17.7	13
	Ti(C,N)+(Ti,Al)N	2.8	3076	39	16.5	15
	Ti(C,N) (2)	2.1	3101	77	13.5	53
	(Al,Ti)N	2.5	3301	100	9.8	55
	(Ti,Al)N	3.5	3327	109	20.9	60
	(Al,Cr)N	3.8	2867	96	27.2	45
	Ti(C,N)+Al_2O_3+TiN	8.4	2315	93	250.7 1)	23
					421 2)	
	Ti(C,N)+TiN	1.8	2443	110	356 1)	27
					294.5 3)	
Sialon tool ceramics	uncoated	-	2035	-	-	11
	Ti(B,N)	1.3	2676	13	57	5
	(Ti,Zr)N	2.3	2916	21	13.6	5,5
	Ti(C,N) (1)	1.5	2872	25	21.3	5
	Ti(C,N)+(Ti,Al)N	1.4	2786	36	24	6
	Ti(C,N) (2)	1.8	2843	26	18.7	9
	(Al,Ti)N	3.0	3600	112	8.2	72
	(Ti,Al)N	5.0	2961	21	40	9
	(Al,Cr)N	4.8	2230	53	16.7	50
	Ti(C,N)+Al_2O_3+TiN	7.0	2669	43	266.5 1)	3
					324 2)	
	Ti(C,N)+TiN	1.3	2746	72	332 1)	15
					112 3)	

1) TiN layer; 2) Al_2O_3 layer; 3) Ti(C,N) layer

It is important that the presented here research ranks the coatings with respect to the exploitative durability of cutting edges made of sintered carbides and sialon ceramics covered with the investigated coatings. The durability of cutting edges (Table 1) made of sialon tool ceramics covered with the investigated coatings is within the range from 5 to 72 minutes of cutting trials of gray cast iron, and the durability of the cutting edges made of sintered carbides with the investigated coatings is within the range from 13 to 60 minutes. It should be also emphasized here that the highest exploitative durability is exhibited by cutting edged made of sialon ceramics covered with the coatings (Al,Ti)N and (Al,Cr)N, and the durability of non-coated sialon cutting edges is 11 minutes. In the case of sintered carbide cutting edges, the highest exploitative durability is exhibited by the inserts coated by (Ti,Al)N and (Al,Ti)N, and the durability of sintered carbide inserts without coating was estimated at 2 minutes of cutting trials.

The critical loading (Table 1) is to a great extent dependent on the applied coating material (chemical composition, phase composition). The said dependence is particularly evident in the case of PVD coatings on sialon ceramics substrate. The coatings in which only phases TiN and Ti(C,N) are present demonstrate low adhesion to the sialon substrate L_c =13÷36N. And the coatings containing the phase AlN are characterized by a very good adhesion to the substrate L_c =53÷112N. It should be underlined that sialons belong to covalence ceramics, whereas in the coatings containing isomorphic phases with titanium nitride TiN, metallic bonds occur, which results in low adhesion of these coatings to the substrate of a different bonding. In the case of coatings containing the AlN phase of the hexagonal lattice, there occur covalence bonds, in the analogous manner as in the ceramic substrate, which, in effect, yields good adhesion of these coatings to the substrate. It bespeaks of the fact that the type of interatomic bonds occurring in the material of substrate and coating exerts a great influence on the adhesion of the coatings to the substrate.

The adherence of coatings to the substrate from sintered carbides, apart from adhesion, is also dependent on slight diffusive dislocation of chemical elements in the contact zone, which is the result of the implantation of high-energy ions falling down on the negatively polarized substrates. This has been confirmed by the research results obtained with the application of glow discharge optical spectrometer GDOES (Fig. 2), since the high-energy ions falling down on the polarized substrate bring about numerous phenomena, among others local temperature rise, faster chemisorption, intensification of surface diffusion processes and into the substrate. There may also occur a slight penetration of ions (about several nm) and partial sputtering of atoms of the coating being deposited.

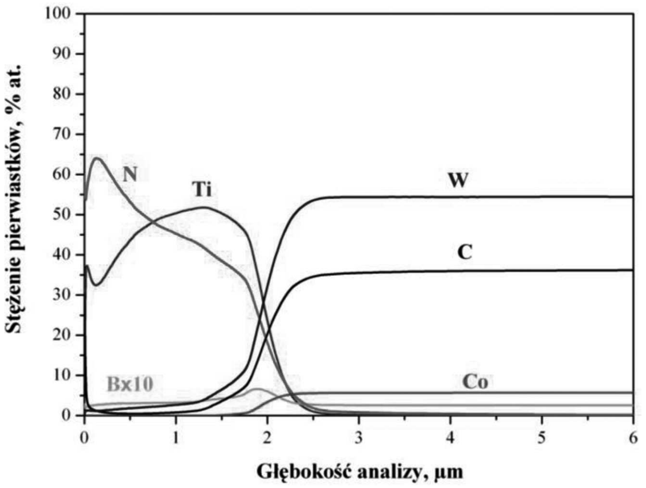

Fig. 2. Changes of constituent concentration of the Ti(B,N) coating and the sintered carbides substrate material

Also the observed structures of thin foils obtained from the investigated coatings bespeak of the fact that the coatings are characterized by high grain-fineness. Furthermore, the diffractive tests on thin foils confirm the presence of isomorphic phases with titanium nitride of the cubic lattice and AlN phase of the hexagonal lattice in the case of (Al,Ti)N coating (Fig. 3).

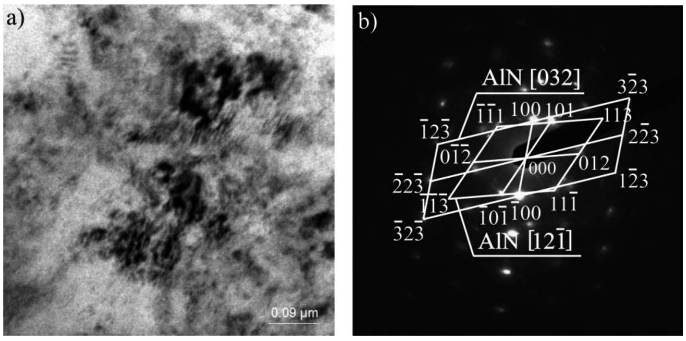

Fig. 3. Structure of the thin foil from the (Al,Ti)N coating: a) bright field; b) diffraction pattern from area a and solution of the diffraction pattern

Basing on the fractographic research it was found that the coatings demonstrate compact structure without pores or non-continuities, similar to the column structure corresponding to zone IV (T) according to Thornton's model. And the coatings Ti(B,N), (Ti,Zr)N and (Ti,Al)N obtained on sialon ceramics have the structure of slightly bigger column grains, close to zone II according to Thornton's model (Fig. 4). Moreover, the particular layers of the multi-layer coatings demonstrate compact structure without delamination or defects and they closely adhere to one another.

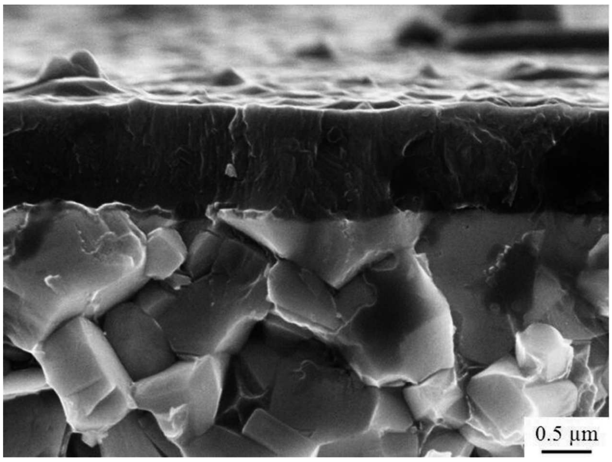

Fig. 4. Fracture of the Ti(B,N) coating deposited onto the sintered carbides substrate

In effect of the microhardness tests it was found that the microhardness range for the coatings obtained on sialon ceramics is within 2315÷3301 HV, and the microhardness range for the coatings obtained on sintered carbides is within 2230÷3600 HV (Table 1).

Basing on the experimental set of results, an artificial neural network was elaborated which allows to determine if there is a dependency between the properties of the coatings such as hardness, adhesion to substrate, grain size or thickness and cutting ability of cutting edges covered by the investigated coatings. The value of mean absolute error, standard deviation and Pearson's correlation factor for the training, validation and testing sets bespeak of the fact that the applied artificial neural networks correctly reflect the modeled relations (Table 2).

TABLE 2

Regression statistics of artificial neural network trained for prediction of PVD and CVD coatings properties deposited onto sialon ceramics

Network architecture	**Regression statistics**	**Data sets**		
		Training Set	**Validation Set**	**Testing Set**
MLP3 4:4-6-1:1	Average absolute error	2.57	2.17	2.74
	Standard deviation ratio	0.14	0.10	0.19
	Pearson correlation	0.99	0.99	0.98

The sensitivity analysis of input data on output data shows that the durability of the cutting edge is principally dependent on the adhesion of the coatings to the substrate (Table 3, Fig. 5). The change of critical loading, being the measure of coatings' adhesion, influences the change of cutting edge durability to the highest extent. The other properties like microhardness, coating thickness and grain size have lower impact on the durability changes of the investigated cutting edges. It should be emphasized, however, that from among the other properties the change of grain size has the highest impact on the durability changes of the investigated cutting edges, in particular in the case of coated sialon ceramics; at the same time the durability of the cutting edges is reversely proportional to grain size. The change of microhardness or thickness of the investigated coatings has only slight influence on the durability changes of the investigated cutting edges.

TABLE 3

Results of sensitivity analysis of input data for output data of artificial neural network trained for prediction of PVD and CVD coatings properties deposited onto sialon tool ceramics

Data sets	Statistics	Micro-hardness	Critical load Lc	Grain size	Thickness
Training	Range	4	1	2	3
	Error	2.78	20.30	18.45	5.11
	Ratio	1.33	9.71	8.82	2.44
Validation	Range	4	1	2	3
	Error	3.08	27.12	15.26	4.89
	Ratio	2.49	21.96	12.36	3.96

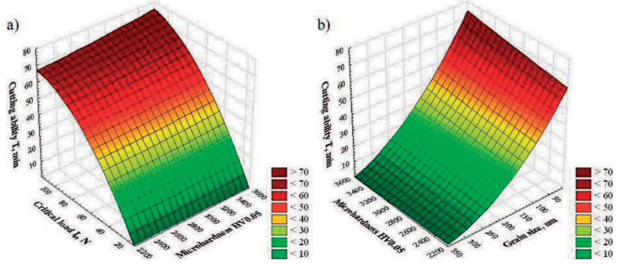

Fig. 5. a) Evaluation of the PVD and CVD coatings critical load and the microhardness influence of tool life T for sialon ceramics tools coated with PVD and CVD coatings determined by artificial neural networks at a fixed coating thickness 3.0 microns and particle size 8.2 nm, b) Evaluation of the PVD and CVD coatings the microhardness and grain size influence of tool life T for sialon ceramics coated with PVD and CVD coatings determined by artificial neural networks at a fixed coating thickness 3.0 microns and critical load 105 N

5. Conclusions

Basing on the carried out research studies the following conclusions can be formulated:

1. It was found that the exploitative durability of the cutting edges from sialon ceramics and sintered carbides covered by gradient and multilayer PVD and CVD coatings depends principally on the adhesion of the coatings to substrate, and to a lower degree on grain size in the coatings, their thickness or hardness changes within 2300÷3500HV.
2. The type of substrate on which the coating is deposited has the influence on the microstructure of the obtained coatings. The PVD coatings of the isomorphic structure with titanium nitride TiN deposited on the substrates from sintered carbides usually demonstrate smaller grains and higher thickness as compared to their deposition on sialon ceramics. Negatively polarized substrate from sintered carbides brings about the situation where ions which reach the substrate gain higher kinetic energy, the temperature increases to a greater extent than in the case of non-polarized substrate, surface mobility of atoms is rising which leads to the generation of new nuclei and refinement of structure, and furthermore, the growth rate of the coatings on the polarized substrate is higher than on the non-polarized substrate.
3. The PVD coatings demonstrate good adhesion to the substrate from sintered carbides, they are characterized by adhesive-diffusive adhesion mechanism which is conditioned by the diffusive dislocation of chemical elements in the transit zone coating-substrate, which in the case of PVD coatings is effected by the implantation of high-energy ions falling on negatively polarized substrate, and in the case of CVD coatings results in the situation where both the working gas and carbon coming from the substrate are the source of carbon. And good adhesion to sialon ceramics is exhibited by PVD coatings containing AlN phase of the hexagonal structure having covalence bonds of the same type as ceramic substrate.

REFERENCES

[1] D. Pakuła, L.A. Dobrzański, A. Križ, M. Staszuk, Investigation of PVD coatings deposited on the Si3N4 and sialon tool ceramics, Archives of Materials Science and Engineering **46**, 1, 53-60 (2010).
[2] L.A. Dobrzański, M. Staszuk, K. Gołombek, A. Śliwa, M. Pancielejko, Structure and properties PVD and CVD coatings deposited onto edges of sintered cutting tools, Archives of Metallurgy and Materials **55**, 1, 187-193 (2010).
[3] L. Cunha, L. Rebouta, F. Vaz, M. Staszuk, S. Malara, J. Barbosa, P. Carvalho, (...), J.P. Riviere, Effect of thermal treatments on the structure of MoNxOy thin films, Vacuum **82**, 12, 1428-1432 (2008).
[4] M. Soković, J. Kopač, L.A. Dobrzański, J. Mikuła, K. Gołombek, D. Pakuła, Cutting characteristics of PVD and CVD – Coated ceramic tool inserts, Tribology in Industry **28**, 1-2, 3-8 (2006).
[5] L.A. Dobrzański, D. Pakuła, Comparison of the structure and properties of the PVD and CVD coatings deposited on nitride tool ceramics, Journal of Materials Processing Technology **164-165**, 832-842 (2005).
[6] L.A. Dobrzański, D. Pakuła, A. Křiž, M. Soković, J. Kopač, Tribological properties of the PVD and CVD coatings deposited onto the nitride tool ceramics, Journal of Materials Processing Technology **175**, 179-185 (2006).
[7] R.P. Martinho, F.J.G. Silva, A.P.M. Baptista, Cutting forces and wear analysis of Si3N4 diamond coated tools in high speed machining, Vacuum **82**, 1415-1420 (2008).
[8] G. Castro, F.A. Almeida, F.J. Oliveira, A.J.S. Fernandes, J. Sacramento, R.F. Silva, Dry machining of silicon-aluminium alloys with CVD diamond brazed and directly coated Si3N4 ceramic tools, Vacuum **82**, 1407-1410 (2008).

[9] M.W. Richert, A. Mazurkiewicz, J.A. Smolik, The deposition of WC-Co coatings by EBPVD technique, Archives of Metallurgy and Materials **57**, 2, 511-516 (2012).

[10] M. Pancielejko, W. Precht, Structure, chemical and phase composition of hard titanium carbon nitride coatings deposited on HS 6-5-2 steel, Journal of Materials Processing Technology **157-158**, 394-398 (2004).

[11] K. Lukaszkowicz, J. Szewczenko, M. Pancielejko, Structure, mechanical properties and corrosion resistance of PVD gradient coatings deposited onto the X40CrMoV5-1 hot work tool steel substrate, International Journal of Materials and Product Technology **39**(1/2), 148-158 (2010).

[12] M. Nalbant, H. Gökkaya, İ. Toktaş, Gökhan Sur, The experimental investigation of the effects of uncoated, PVD- and CVD-coated cemented carbide inserts and cutting parameters on surface roughness in CNC turning and its prediction using artificial neural networks, Robotics and Computer-Integrated Manufacturing **25**, 211-223 (2009).

[13] M. Reza Soleymany Yazdi, A. Mahyar Khorasani, Mehdi Faraji, Optimization of coating variables for hardness of industrial tools by using artificial neural networks, Expert Systems with Applications **38**, 12116-12127 (2011).

[14] E.O. Ezugwu, D.A. Fadare, J. Bonney, R.B. Da Silva, W.F. Sales, Modelling the correlation between cutting and process parameters in high-speed machining of Inconel 718 alloy using an artificial neural network, International Journal of Machine Tools & Manufacture **45**, 1375-1385 (2005).

[15] Ihsan Korkut, Adem Acır, Mehmet Boy, Application of regression and artificial neural network analysis in modelling of tool-chip interface temperature in machining, Expert Systems with Applications **38**, 11651-11656 (2011).

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