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Gradient PVD coatings deposited on the sintered tool materials

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ABSTRACT

Purpose: The paper presents investigation results of properties of the sintered tool materials: cemented carbides, cermets and Al_2O_3 type oxide tool ceramics with gradient (Ti,Al)N and Ti(C,N) coatings deposited with the cathodic arc evaporation CAE-PVD method.

Design/methodology/approach: Analysis of the mechanical and functional properties: surface roughness, microhardness, adhesion, and wear. Analysis of the structure (SEM). Computer simulation of stresses carried out in ANSYS environment, using the FEM method.

Findings: The wear resistant gradient coatings of the type (Ti,Al)N and Ti(C,N) deposited on the investigated sintered tool materials yield a considerable increase of microhardness in the surface area, which, combined with good adhesion of the coating to the substrate, obtained in effect of the application of gradient structure of the coating, has the influence on the applicability properties of these materials during machining tests. A more advantageous distribution of stresses in gradient coatings influence on better mechanical properties. The distribution of stresses on the coating surface has the influence on microhardness, while distribution of stresses in the contact area between the coating and substrate has the influence on the adhesion of coatings.

Practical implications: Deposition of hard, thin, gradient coatings on materials surface by PVD method features one of the most intensely developed directions of improvement of the working properties of materials.

Originality/value: Techniques of gradient coatings deposition is one of the most spectacular aspect of the materials engineering development in the last years. Uunique combination of substrates and coatings is presented in the paper.

Keywords: Tool materials; Gradient coatings; PVD; Stresses

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PROPERTIES

1. Introduction

Cemented carbides and cermets belong to a class of hard, wear-resistant too materials in which the hard carbide particles are bound together, or cemented, by a ductile metal binder. Ceramic materials with high hardness and high strength in the broad range of working temperatures and with low abrasion wear, with the Al_2O_3 based ones among them, are used more and more often for cutting tools [1-4].

The improvement of the functionality properties of the tools and the reduction of ecological hazards can be effected through the application of the technology of hard gradient coatings deposited on the tools in physical vapour deposition processes, principally by ensuring better conditions of tribological contact in the machining area and by eliminating the cutting tool lubricants. The machining process is becoming so common that it is necessary to intensify research studies concerning not only the selection of appropriate material for tools but also the deposition technology of modern wear resistant coatings, primarily such as gradient coatings, and to elaborate them and verify in industrial conditions. The application of PVD for the acquisition of gradient coatings of high wear resistance, also in high temperatures, enables to improve the properties of these materials in machining conditions, among others by the reduction of friction factor, increase of microhardness, improvement of tribological contact conditions in the contact area tool-machined item it makes it also possible to protect these materials against adhesive or diffusive wear and against oxidation [4, 17-25, 27].

At the present time, coatings obtained by PVD process are widely used in the sintered tool materials industry. Coatings based on (Ti,Al)N as well as Ti(C,N) were developed to provide better performance over titanium nitride since the incorporation of aluminum or carbon atoms into TiN is conducive to greater hardness and smaller coefficient of friction of the coatings [5-16, 19].

The main objective of the present paper is to investigate the properties of sintered tool materials, including cemented carbides, cermets and Al_2O_3+TiC type oxide tool ceramics deposited with gradient (Ti,Al)N and Ti(C,N) coatings in the cathodic arc evaporation CAE-PVD method.

2. Methodology of research

The research studies were carried out on sintered tool materials, such as cemented carbides, cermets and oxide ceramics, deposited and non-deposited with gradient (Ti,Al)N and Ti(C,N) were resistant coatings, using the cathodic arc evaporation method (CAE). The characteristics of the investigated materials are presented in Table 1.

The PVD deposition process of gradient (Ti,Al)N and Ti(C,N) coatings was carried out in the Institute of Engineering Materials and Biomaterials of the Silesian University of Technology at Gliwice, on the apparatus DREVA ARC400 of the German Company VTD Vakuumtechnik. The apparatus is equipped with three independent sources of metal vapours. Before the deposition of coatings, the substrates were prepared for the deposition. The preparation process consisted of two stages. The first stage was carried out outside the operating chamber of the coating apparatus. The multi-point inserts were subjected to chemical cleaning, using washing and rinsing in ultrasonic washers and cascade cleaners, and then they were dried in the stream of hot air. The second preparation stage was carried out in the vacuum chamber of the PVD coating apparatus. That stage consisted in heating the substrate to the temperature of around 400°C with a beam of electrons emitted from the hollow cathode in argon atmosphere with lowered pressure, and then in ionic cleaning using Ar ions with the polarization voltage of the substrate of -300 V for 25 minutes. For the deposition of coatings, shields of the diameter of 65 mm cooled with water were applied. The shields contained pure Ti and the alloy TiAl of 50:50% at. The vacuum of 10^{-4} Pa was created in the operating chamber. The coatings were deposited in the atmosphere of inert gas Ar and reactive gases N_2 in order to obtain nitrides, and the mixture of N_2 and C_2H_2 to obtain carbonitride coatings. The gradient concentration change of the chemical composition along the cross-section of the coatings was obtained by changing the dosage proportion of the reactive gases or by changing the intensity of evaporation current of the shield on arc sources.

Observations of surfaces and structures of the deposited coatings were carried out on the transverse fractures in the scanning electron microscope SUPRA 35. To obtain the fracture images the Secondary Electrons (SE) detection method has been used with the accelerating voltage in the range of 15-20 kV and maximum magnification 60 000 x.

Table 1.
Characteristics of the investigated materials

Substrate	Coating	Coating thickness, μm	Roughness, R_a, μm	Microhardness, HV	Critical Load, L_c, N	Tool life t, min
Cemented carbide*	uncoated	-	0.13	1755	-	2.5
	(Ti,Al)N	2.6	0.14	3000	56	25.5
	Ti(C,N)	2.7	0.11	2850	64	5.0
Cermet**	uncoated	-	0.06	1850	-	2.5
	(Ti,Al)N	3.0	0.12	3150	63	22.0
	Ti(C,N)	2.6	0.11	2950	60	9.5
Al_2O_3+TiC***	uncoated	-	0.10	2105	-	12.5
	(Ti,Al)N	3.2	0.24	3200	65	40
	Ti(C,N)	2.1	0.21	2950	55	19

***** phase composition: WC, TiC, TaC, Co,
** phase composition: TiCN, WC, TiC, TaC, Co, Ni,
*** phase composition: Al_2O_3, TiC.

The thickness of the coatings was tested using the calotest method which consists in the measurement of the characteristic quantities of the crater effected by the wear on the surface of the investigated specimen brought about by a steel ball 20 mm in diameter. The space between the rotating ball and specimen surface was being fed with the suspension of diamond grains of the diameter of 1 µm. The test time was accepted at 120 seconds. The measurement of wear extent was carried through the observations on the illumination metallographic microscope LEICA MEF4A. In order to obtain average thickness values of the measured coatings, 5 measurements were carried out for each of the investigated specimens. Additionally, to verify the obtained results, the measurements of coating thickness were carried out in the scanning electron microscope at the transverse fractures of the specimens.

The measurements of the surface roughness of the polished specimens without coatings and with deposited coatings were measured in two mutually perpendicular directions on the profilometer Surftee 3+ of Taylor Hobson Company. The accepted measurement length was l=0.8 mm, and measurement accuracy ±0.02 µm. Additionally, to confirm the obtained results, the roughness measurements of specimen surfaces were carried out on the confocal microscope LSM 5 Exciter of Zeiss Company. The parameter R_a was accepted as the quantity describing the surface roughness, in compliance with the Standard PN-EN ISO 4287:1999.

The hardness of the investigated materials was determined using the Vickers method. The hardness of the deposited substrates from sintered tool materials was tested using the Vickers method with the load of 2.94 N (HV 0.3) in compliance with the Standard PN-EN ISO 6507-1:2007. The tests on microhardness of the deposited coatings were performed on the microhardness meter Future Tech, making use of the Vickers dynamic method. We applied the load of 0.98 N (HV 0.1), enabling, to the highest possible extent, to eliminate the influence of substrate on the obtained results. The measurements were carried out in the mode of periodic loading and unloading, in which the tester loads the indenter with the preset force, maintains the load over some time period and then unloads it. The trial makes it possible to observe the changes of plastic and elastic strain of the investigated material, respectively during the loading and unloading due to a high-precision measurement system which can record the depth of the formed imprint in successive phases of the test. The measurements were carried out making 6 imprints for each of the investigated specimens. An average was determined, as well as standard deviation and confidence interval, assuming the confidence factor at $1-\alpha = 0.95$.

The adhesion assessment of the deposited coatings to the investigated sintered tool materials was carried out using the scratch-test on the apparatus REVETST of CSEM Company. The method consists in moving the diamond indenter along the surface at constant speed, with the loading force increasing proportionally with the movement. The tests were carried out for the loading force within the range of 0±100 N, increasing with the speed of (dL/dt)=100 N/min along the path of 10 mm.

The critical load Lc at which the coating loses its adhesion was determined basing on the value of acoustic emission (AE) recorded during the measurement and on the observation of scratch lines effected during the scratch-test. The character of the fault was assessed basing on the observations in the scanning electron microscope Zeiss Supra 35 and in the confocal microscope LSM 5 Exciter of Zeiss Company.

The operating properties of the deposited coatings were determined basing on the technological machining trials at room temperature. The tests on cutting ability of the investigated tool materials without coatings and with the deposited coatings were carried out basing on the technological cutting trials without cutting tool lubricants on a universal numerically controlled lathe Gildemeister NEF 320. The cast iron EN-GJL-250 of the hardness of around 250 HV was selected as material subjected to machining. For the technological cutting trials, we applied inserts fixed in a universal lathe chuck which ensures the maintenance of geometric parameters of the inserts (Fig. 1).

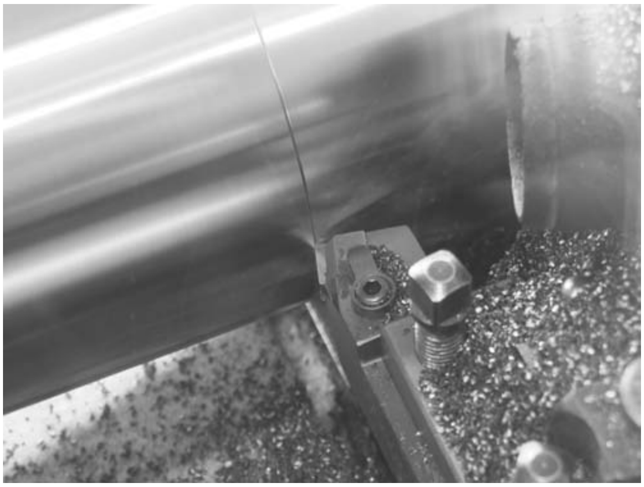


Fig. 1. Machining test overview

The following parameters were accepted for the cutting ability tests:

- rate of feed f = 0.1 mm/rev.,
- turning depth ap = 1 mm,
- cutting velocity vc = 150 m/min.

The durability of the inserts was determined basing on the measurements of wear strip width on the tool flank, measuring the average wear strip width VB after the machining in a definite time interval. The machining trials were being stopped when the VB value exceeded the accepted criterion for after-machining, i.e. VB = 0.2 mm. In the case of non-deposited tools, the trial was being carried out until the wear criterion had been reached , and the duration of the trial for the tools with deposited coatings was the same or longer than in the case of non-deposited tools, whereby we can compare the wear strip width VB after the wear criterion has been reached by the non-deposited specimen. The VB measurements were carried out with the application of the illumination microscope Carl Zeiss Jena. The images of tool flank and attack surface of the inserts of different wear degree as well as the topography of the fractured tool with the use of a 3D model were obtained with the application of the scanning electron microscope Zeiss Supra 35 and of the confocal microscope LSM 5 Exciter of Zeiss Company. The analyses of chemical composition in the microareas were carried out using the EDS method. The obtained research results were presented in the form of graphs determining the dependence of wear strip width on the tool flank VB as the function of testing time, assuming the preset conditions of the experiment.

The work presents the application of the finite elements method for the analysis of the distribution of internal stresses in the coatings obtained in the PVD process, as dependent on the parameters of the process and the material of the substrate and coating. The model where of objective is to determine the internal stresses in gradient coatings (Ti,Al)N and Ti(C,N) on the substrate from cemented carbides, cermet and oxide tool ceramics, was elaborated using the finite elements method, assuming true dimensions of the specimen. The geometry of the insert with the deposited gradient coatings as well as the calculations were carried out using the program ANSYS 12.0. On account of the predicted simulation range, parametric calculation files were elaborated which allowed to perform the analysis in a comprehensive way. We employed the experience involving computer simulation works in material engineering carried out for many years at the Division of Materials Processing Technology, Management and Computer Techniques in Materials Science of the Institute of Engineering Materials and Biomaterials of the Silesian University of Technology [26].

Since it was necessary to calculate internal stresses in the material of the chemical composition which was changing in the way perpendicular to the surface, the ideographic differentiation of the modeled gradient coatings was carried out into zones corresponding to the areas of similar chemical composition. The model with the spherical division of gradient coating was elaborated in the way ensuring that it was possible to determine the averaged internal stresses in the coating areas important in view of the applicability properties and to compare the obtained results with the calculations carried out for homogeneous coatings.

The following boundary conditions were accepted to simulate the internal stresses in the gradient (Ti,Al)N and Ti(C,N) coatings on different substrates:

- the temperature change of the PVD process is reflected by cooling the specimen from 500°C to the ambient temperature of 20°C,
- for the coatings (Ti,Al)N and Ti(C,N) and for the substrate from cemented carbides, cermet and oxide tool ceramics, the material properties were accepted basing on literature data [5] and MatWeb catalogue. The discrepancies in literature data involving the values of physical properties of particular materials result from different acquisition methods, from the differences in the structure and composition of the materials and from errors in the applied measurement method [26],
- the substrate of the investigated specimen is immobilized due to depriving all nodes lying on this axis of all degrees of freedom.

3. Results

The investigated sintered tool materials are characterized by well condensed compact structure without pores (Fig. 2), and in the case of oxide ceramics Al_2O_3+TiC the topography of the fracture surface bespeaks of high brittleness [8-9], characteristic of oxide ceramic materials (Fig. 3).

The deposited gradient coatings have a continuous structure. It was demonstrated that the coatings are uniformly deposited and are characterized by close adhesion to the substrate, without pores, cracks and discontinuities (Figs. 4-5).

The observations involving the surface morphology of the coatings fabricated in the PVD-CAE process on the substrate from cemented carbides, cermets and oxide ceramics are indicative of high non-homogeneity connected with the occurrence of numerous droplet-shaped microparticles (Fig. 6).

The observed morphological defects brought about during the deposition of the coating are most probably effected by splashing of titanium droplets liberated from the titanium shield onto the substrate surface, which has been confirmed by EDS tests from the microareas (Fig. 6). The droplets observed in SEM assume regular shapes, their size is different and is within the range from the tenths of a micrometer to around a dozen micrometers. Agglomerates created on the coating surface from several joined microparticles was also observed. Furthermore, hollow areas generated in effect of the liberation of titanium microparticles after the termination of the coating deposition process were observed.

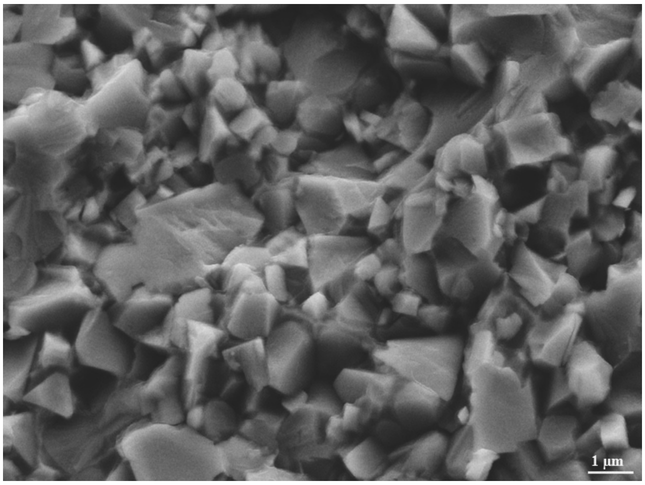


Fig. 2. Structure from the cemented carbides

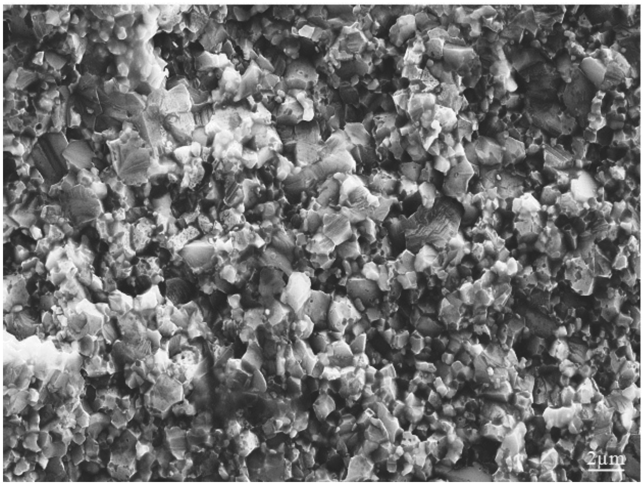


Fig. 3. Structure from the oxide ceramics Al_2O_3+TiC

The deposition of gradient coatings wear resistant of the type (Ti,Al)N and Ti(C,N) on the investigated sintered tool materials results in the increase of roughness parameter R_a which is within the range from 0.11-0.24 μm, and is higher than in the case of material surfaces without coatings (Table 1). The roughness increase of the surfaces of the deposited coatings should be linked to the character of PVD process, which was confirmed by the morphological tests of the surface in the scanning electron microscope (Fig. 6).

The deposited PVD coatings are characterized by good adhesion to the substrate within the range L_c=55-65N (Figs. 7-9). In general, the deposition of wear resistant gradient (Ti,Al)N and Ti(C,N) coatings on the investigated sintered tool materials results in a considerable increase of microhardness in surface area, which, combined with the good adhesion of the coating to the substrate obtained in effect of the application of gradient structure of the coating, yields good functionality properties of these materials, confirmed during machining tests (Table 1).

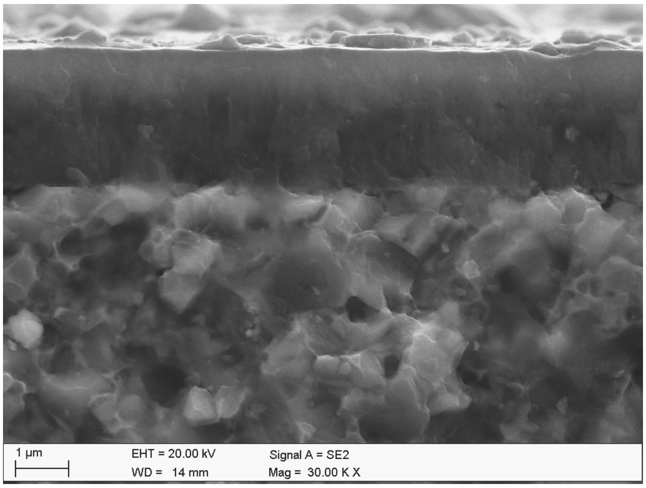


Fig. 4. Fracture surface of the gradient Ti(C,N) coating deposited onto the cermets substrate

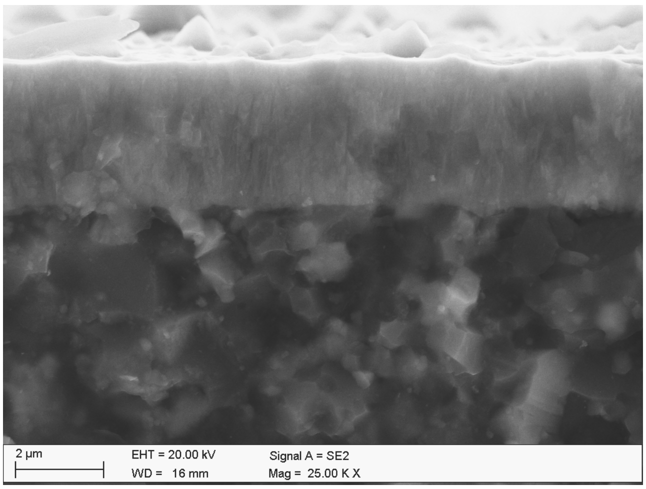
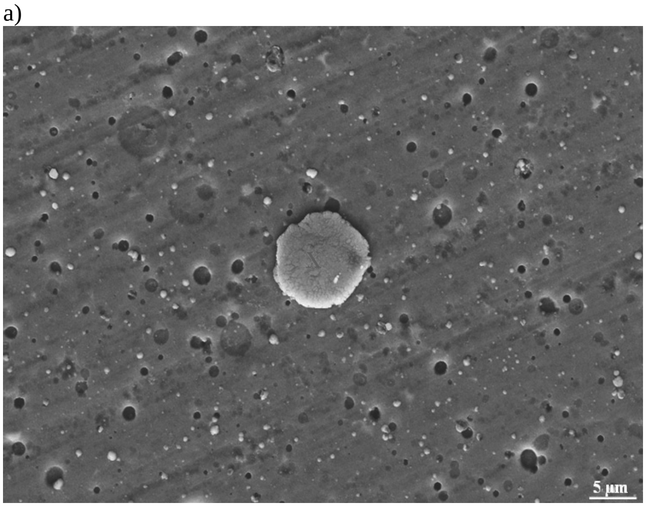


Fig. 5. Fracture surface of the gradient (Ti,Al)N coating deposited onto the Al_2O_3+TiC oxide tool ceramics substrate

The hardness of the substrate material is 1755 HV for cemented carbides, 1850 HV for cermets and 2105 HV for oxide ceramics (Table 1). The deposition of the coatings (Ti,Al)N and Ti(C,N) on the investigated sintered tool materials results in a considerable increase of microhardness in the area around the surface within the range of 2850-3200 HV (Fig. 10).

Hardness depends on the values of intermetallic bonds, so the hardest materials have covalence bonds, and the increase of the share of ionic character of the bond is associated with the drop of hardness [5]. Basing on the carried out research it was demonstrated that the hardness of Ti(C,N) coatings, in which the metallic phases TiN and TiC occur, demonstrates lower hardness than (Ti,Al)N coatings in which there are both metallic bonds TiN and covalence bonds AlN. The deposition of the wear resistant coatings on the investigated substrates results in a considerable increase of microhardness of the surface layer, which contributes to lower wear intensity of the cutting edge of machining tools from cemented carbides, cermets and oxide ceramics during the machining process.

a)



b)

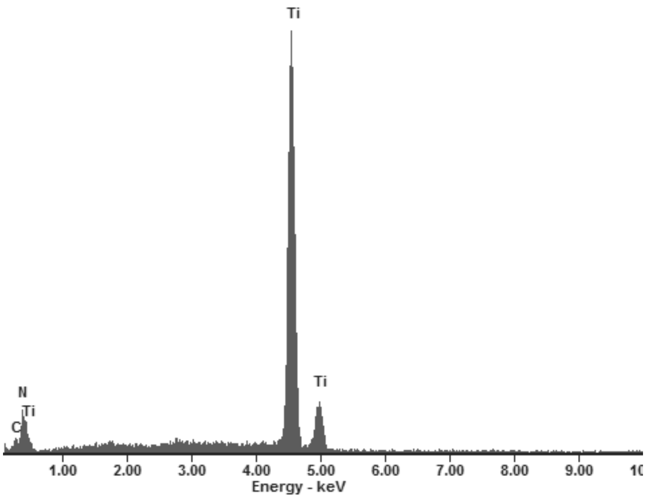


Fig. 6. a) Surface topography of the gradient Ti(C,N) coating deposited onto the cermet substrate and b) X-ray energy dispersive plot the area X

In order to check the correlation between the hardness of the investigated materials (Fig. 10) and the functional properties of the multi-point inserts in the machining tests (Fig. 11) were carried out, the durability of the inserts was determined on the basis of the measurement of wear width on the flank face after the machining in a definite time interval (Table 1).

The carried out research confirmed that better results are obtained by the tools deposited with (Ti,Al)N coatings, independent of the substrate material. It is connected among others with high microhardness of the coatings and with high wear resistance in raised temperature of the (Ti,Al)N coating. It was demonstrated that for the oxide ceramics Al_2O_3+TiC deposited with PVD coatings, the longest durability of cutting edges T=40 min is for the gradient coating (Ti,Al)N, as compared to the coatings deposited on cemented carbides and cermets, which can be related with higher hardness of the substrate material (Table 1, Fig. 11).

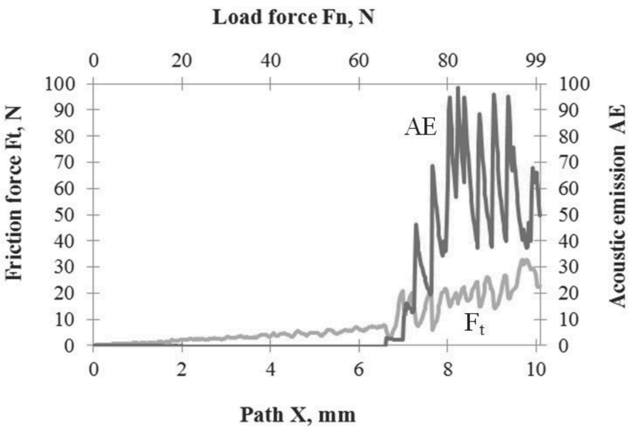


Fig. 7. Diagram of the dependence of the acoustic emission (AE) and friction force F_t on the load for the cemented carbide with the Ti(C,N) coating

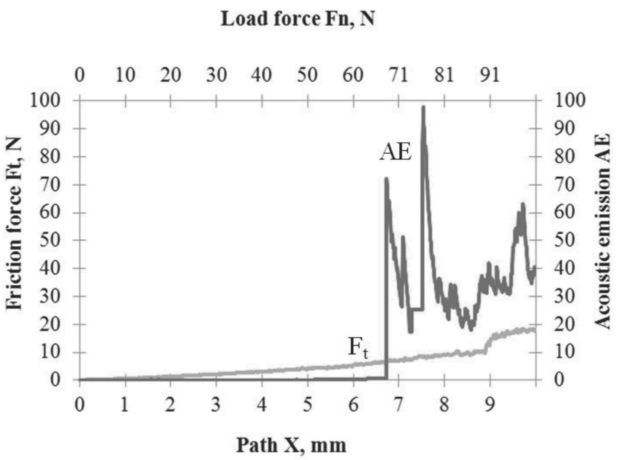


Fig. 8. Diagram of the dependence of the acoustic emission (AE) and friction force F_t on the load for the Al_2O_3+TiC oxide tool ceramics with the (Ti,Al)N coating

In effect of the materialographic observations of the investigated indexable inserts in the scanning electron microscope it was demonstrated that the tools subjected to machining trials show their wear according to abrasive and adhesive mechanism (Fig. 12a). Figure 12b presents X-ray energy dispersive spectrum obtained from substrate material. A build-up of the machined material was found on the tool flank, on the cutting edges from Al_2O_3+TiC oxide tool ceramics with Ti(C,N) coating and was confirmed by the presence of iron reflexes on EDS graphs from the microareas (Fig. 12c). Figure 12d presents EDS measurement of unworn area of Ti(C,N) coating (it corresponds to area "c" on Fig. 12a).

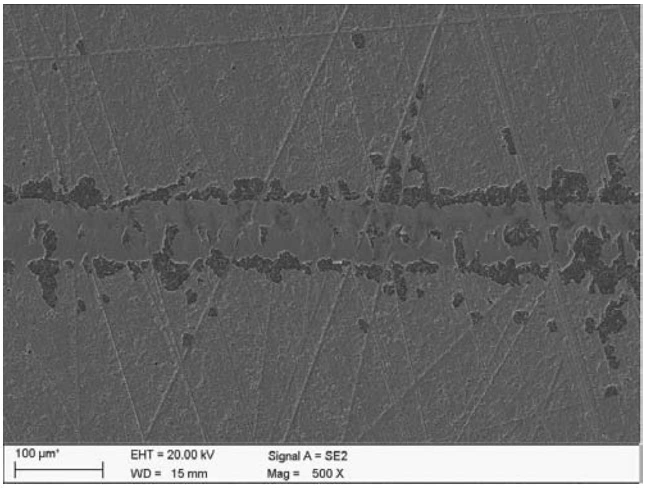


Fig. 9. Characteristic failure obtained by scratch test of the Ti(C,N) coating deposited on Al_2O_3+TiC oxide tool ceramics

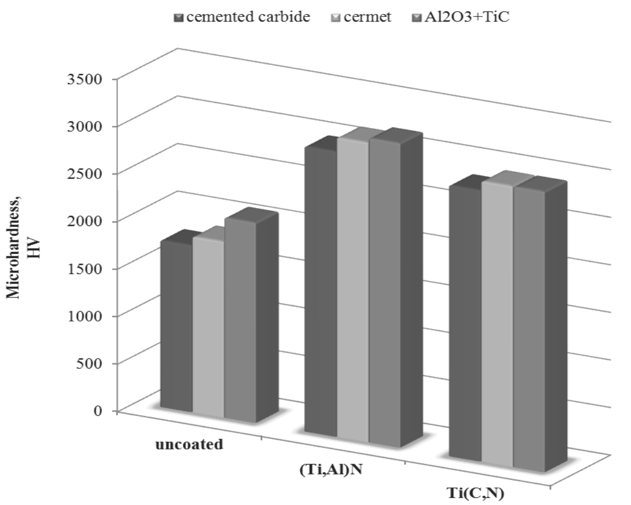


Fig. 10. Comparison of the microhardness of the investigated materials

Due to the application of gradient coatings on all investigated substrate materials, compressive stresses were obtained in the surface layer of the coating having a direct contact with the machined material during the operation process (Table 1). In the case of gradient (Ti,Al)N coatings, a considerable increase of compressive stresses on the coating surface as compared to gradient Ti(C,N) coatings was observed (Fig. 13). The generation of compressive stresses in the surface layer brings about better resistance to cracking, and through the increase of hardness, improves the resistance to wear. The generation of compressive stresses in the surface layer can prevent the formation of cracks when the element in the operational conditions is subjected to stresses generated by external forces.

Yet, an excessive value of compressive stresses can lead to adhesive wear and can bring about the formation of too high tensile stresses under the coating, lowering the fatigue resistance of the element [23]. Volvoda [27] pointed out the relation between the value of stresses and the hardness of the layer of titanium nitrides

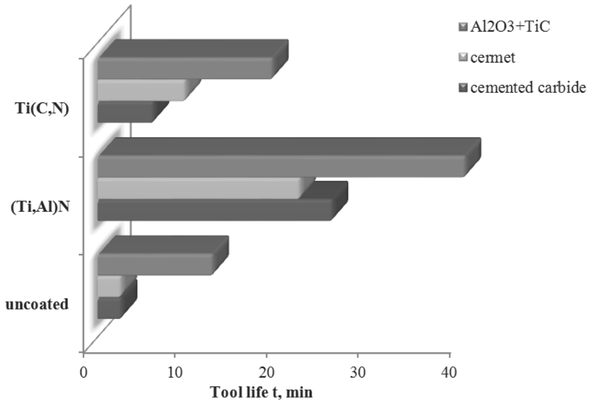


Fig. 11. Comparison of tool life for tools from cemented carbides, cermets and oxide ceramics with gradient (Ti,Al)N and Ti(C,N) type coatings with uncoated tool

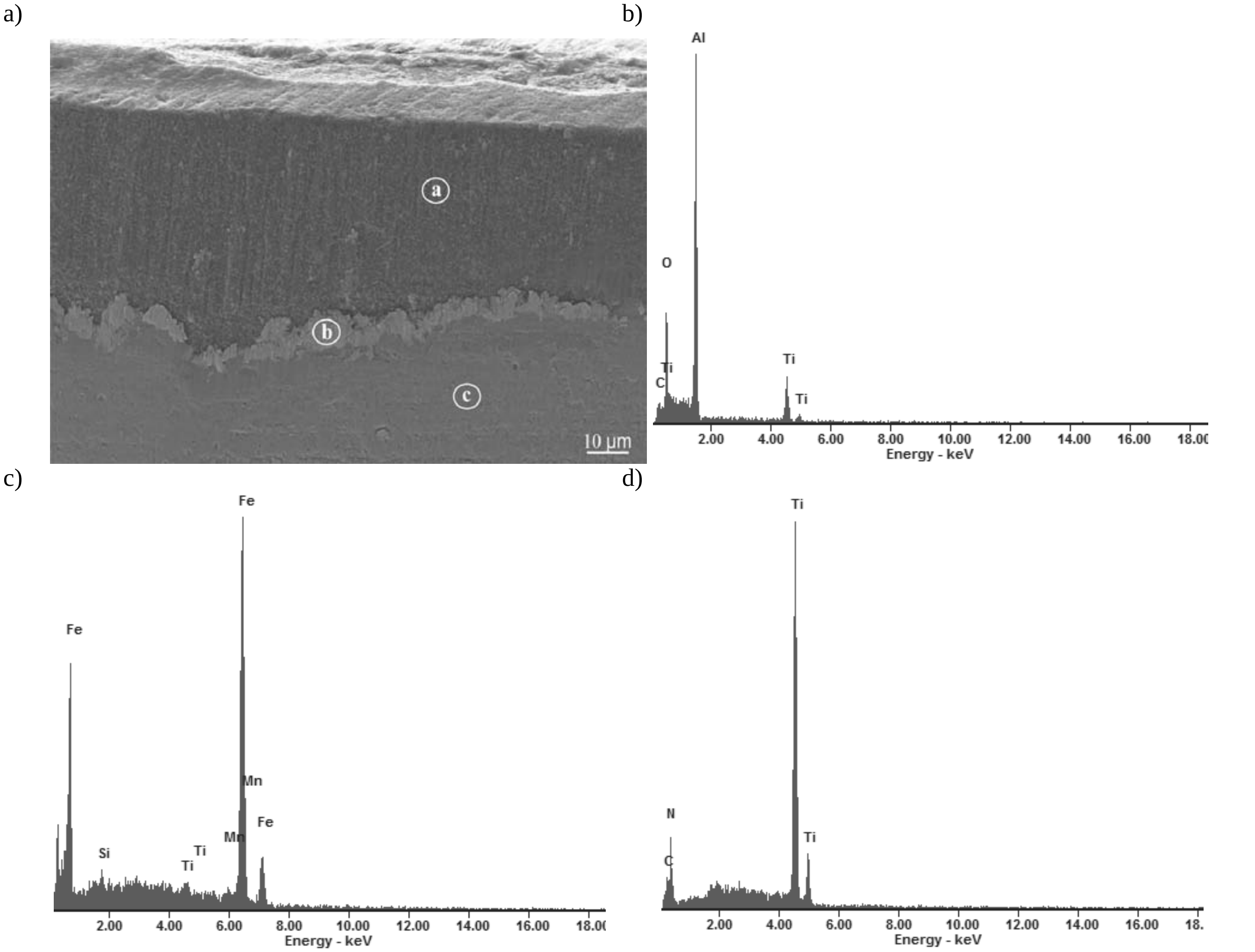


Fig. 12. a) Character of wear of the Al_2O_3+TiC oxide tool ceramics sample with Ti(C,N) coating, investigated with SEM, b) X-ray energy dispersive spectrum from the area "a" on figure a), c) X-ray energy dispersive spectrum from the area "b" on figure a), d) X-ray energy dispersive spectrum from the area "c" on figure a)

obtained in effect of magnetron sputtering, demonstrating that with the increase of compressive stresses the hardness of the obtained layer is progressively increasing. Basing on the carried out research, it was demonstrated that the occurrence of compressive stresses on the surface of gradient coatings of the investigated materials has a positive influence on their mechanical properties, in particular on the microhardness (Table 1).

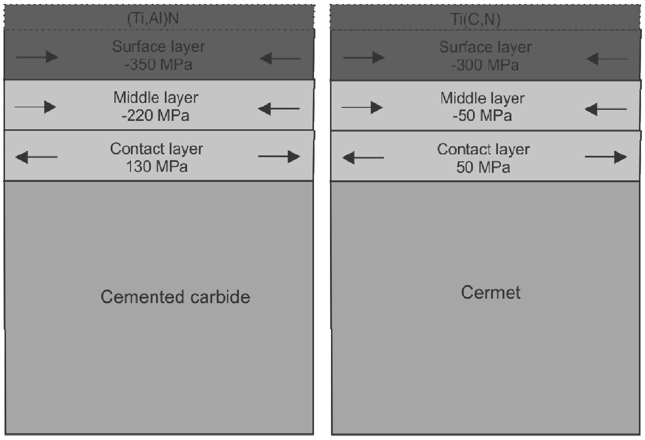


Fig. 13. Schematic distribution of stresses in the gradient coating (Ti,Al)N and Ti(C,N) deposited on cemented carbide and cermet substrate obtained by computer simulation

With the temperature decrease, from the coating deposition temperature (500°C) to the ambient temperature (20°C), internal stresses are generated both in the coating and in the substrate material, connected principally with different thermal expansion of particular materials. The distribution of these stresses is also connected with the geometry of the specimen and with thermal transfer during the cooling process. In effect of non-uniform cooling of the specimen material in the particular areas, the distribution of stresses on the coating surface and their concentration in the corners of the specimen is also non-uniform [23, 26].

4. Conclusions

The results of the investigations of the cemented carbides, cermets and Al_2O_3+TiC type oxide tool ceramics coated with the gradient (Ti,Al)N and Ti(C,N) coatings with use of the cathodic arc evaporation CAE-PVD method are given in the paper.

The roughness of the substrate has the influence on the roughness of the deposited coating and on its structure, as well as on the adhesion of the coating to the substrate. Too high roughness of the substrate (R_a>0.4) can cause a so called thinning out effect and a generation of coatings of low-compacted structure with numerous surface defects and low adhesion to the substrate. With the application of too smooth substrates (R_a<0.04) we can not ensure a satisfying mechanical anchoring of the increasing coating against the substrate [5, 7].

The deposition of the gradient (Ti,Al)N and Ti(C,N) coatings on the investigated sintered tool materials results in a considerable increase of microhardness in the area around the surface. Hardness is a property of material dependant on the values of intermetallic bonds. The hardness of Ti(C,N) coatings, in which the metallic phases TiN and TiC occur, demonstrates lower hardness than (Ti,Al)N coatings in which there are both metallic bonds TiN and covalence bonds AlN. The deposition of the wear resistant coatings on the investigated substrates results in a considerable increase of microhardness of the surface layer, which contributes to lower wear intensity of the cutting edge of machining tools from cemented carbides, cermets and oxide ceramics during the machining tests.

A more advantageous distribution of stresses in gradient coatings influence on better mechanical properties. In particular, the distribution of stresses on the coating surface has the influence on microhardness, while the distribution of stresses in the contact area between the coating and substrate has the influence on the adhesion of coatings.

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INNOVATIVE ECONOMY
NATIONAL COHESION STRATEGY
FORSURF
EUROPEAN UNION
EUROPEAN REGIONAL
DEVELOPMENT FUND

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