Characterisation of the Gradient Coatings TiN/(Ti,Al,Si)N/TiN Type Deposited on Sintered Tool Materials

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The paper presents results of the structural examinations, tests of mechanical and working properties of thin wear resistant gradient coatings of the TiN/(Ti,Al,Si)N/TiN type, deposited in the CAE process onto the substrate from the cermets and cemented carbides. Structural examinations are presented of the applied coatings and their substrate made on the SEM, TEM and on the LM.
Evaluation of the adhesion of the deposited coatings onto the cemented carbides and cermets was made using the scratch test. Cutting properties of the investigated materials were determined basing on the technological continuous cutting tests of the C45E steel. Substrate hardness tests and microhardness tests of the deposited coatings were made on the ultra-micro-hardness tester at 70 mN load. Surface roughness tests were also made before depositing the coatings and after completing the PVD process.

Key words: PVD, gradient coatings, cermets, cemented carbides, cutting ability test, SEM, TEM, scratch test

INTRODUCTION

Fast pace of development of engineering and manufacturing technology brings the necessity for increasing the requirements posed to the contemporary sintered tool materials as regards their working properties. Deposition of hard wear resistance coatings based on carbides, nitrides, or transition metals oxides feature one of the fastest developing directions of research, stimulated by the growing service requirements of machines and equipment, making definite improvement of the sintered tool materials possible (sintered high speed steels, cemented carbides, cermets, ceramics).
Wide choice of coatings available nowadays and technologies for their deposition is an effect of the growing in the last years demand for the state-of-the-art surface modification methods. An increased interest is observed in coatings having joint properties like resistance to tribological wear and corrosion. Tools covered with coatings based on carbides, borides, nitrides, and oxides can work at higher service parameters (temperature, load, etc.). Moreover, the multilayer and multicomponent coatings developed relatively not so long ago make it possible to constitute freely properties of the entire coating as well as of its transition layer, ensuring good adhesion, compensation of the internal stresses, and transmission of the external loads. Tools with such coatings reveal a significant life extension in service compared to the uncoated tools or coated with simple coatings based on mononitrides or carbonitrides, improvement of the tribological contact conditions in the tool-chipmachined material contact zone, and protection of the tool edge from oxidation and extensive overheating. Many aspects pertaining to forming of coatings, including also the process conditions effect on their properties, still remain inexplicable in spite of the enormous interest paid in them by many industrial centres and research laboratories [1-17].

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Therefore, cemented carbides were investigated in this project, coated with the PVD method in the Cathode Arc Evaporation (CAE) with the composite gradient coatings of the TiN/(Ti,Al,Si)N/TiN, and they were compared with the commercially available uncoated tool materials and those coated in the PVD and CVD processes with the single- and multilayer wear resistant coatings.

EXPERIMENTAL PROCEDURE

The tests were carried out on cemented carbides and cermets: uncoated and coated using the PVD method of coatings from the gaseous phase in the Cathodic Arc Evaporation process, with the TiN/(Ti,Al,Si)N/TiN multi-layer wear resistant coatings. The following parameters were used in coating deposition: substrate polarisation -200 V, substrate temperature 550°C, pressure in the chamber 0,2 Pa. Commercial brands of cemented carbides and cermets for similar applications according to the ISO classification, coated both in the PVD or CVD processes, were used for comparative tests. Specifications of the investigated cemented carbides and cermets are presented in Tables 1 and 2.
The roughness measurements of the developed coatings and the substrate were made in two orthogonal directions on the Taylor-Hobson Sutronic3+ device. According to Polish Standard PN EN ISO 4287 the R_a parameter was assumed to be the value describing surface roughness.
Evaluation of the phase composition of the investigated coatings was made using the DRON 2.0 Xray diffractometer, using the filtered cobalt lamp rays with the voltage of 40 kV and heater current of 20 mA. Measurements were made within the 2_ angle range from 30-115°.
The examinations of thin foils were made on the JEOL 3010CX TEM at the accelerating voltage of 300 kV and maximum magnifications 250 000x. The diffraction patterns from the transmission electron microscope were solved using the “Index” computer program.
The metallographic examinations of the substrate and the deposited coatings were made on the LEICA MEF4A light microscope. Observations of surfaces and structures of the developed coatings were carried out on the transverse fractures on the Philips XL-30 SEM. To obtain the fracture ima-

Designation	Coating Type	Coating Composition	Coating thickness, μm	Process type
CC1	–	uncoated	–	–
CER1	–	uncoated	–	–
CC3	monolayer	TiN	2.0	PVD
CC2 CER2	gradient layer	TiN/(Ti,Al,Si)N/TiN	4.0 4.0	PVD PVD
CC4 CER3	multilayer	$TiCN/Al_2O_3$ /TiN TiN/TiC/TiN	10.0 5.0	CVD PVD

Table 1 – Specification of the investigated materials.

Tab.1 – Elenco e caratteristiche dei materiali esaminati.

Designation	Mass concentration of elements, %								
	C	N	Ti	Ta	Ni	Co	W	Nb	Zr
CC1, CC2 (cemented carbide)	0,40	–	5,00	7,00	–	5,80	81,80	–	–
CC3 (commercial cemented carbide)	0,75	–	–	–	–	6,75	92,50	–	–
CC4 (commercial cemented carbide)	1,15	–	–	–	–	8,50	80,00	5,00	5,35
CER1, CER2 (cermet)	0,80	2,00	48,70	13,50	4,70	8,60	21,65	–	–
CER3 (commercial cermet)	1,65	2,40	47,50	10,70	8,80	8,00	20,95	–	–

Table 2 – Chemical composition of the investigated materials (substrate).

Tab. 2 – Composizione chimica del materiale esaminato (substrato).

ges the Secondary Electrons (SE) and the Back Scattered Electrons (BSE) detection methods were used with the accelerating voltage in the range of 15-20 kV.
The microhardness tests using the Vickers method were made on the Shimadzu DUH 202 tester.
The load of 70 mN was employed, making it possible to eliminate to the greatest extent the influence of the substrate material on the measurement results.
Tests of the coatings' adhesion to the substrate material were made using the scratch test, routinely employed in case of the coatings obtained in processes of physical deposition from the gaseous phase. Tests were made on the CSEM Revetest device with the following test conditions: load range 0-200 N, load increase rate (dL/dt) 100 N/min, indenter speed (dx/dt) 10 mm/min, sensitivity of the acoustic emission detector AE 1.2. The character of the developed defect was assessed basing on examinations on the SEM.
The working properties of the developed coatings were determined basin on the technological cutting tests carried out at room temperature. Cutting ability tests of the investigated high speed steels were carried out as the continuous dry turning on the HH V630N lathe with 30 kW power rating. The machined material is the C45E steel quenched and tempered. Multipoint SNMG 120408 inserts were used for the continuous turning, fixed in a universal tool post preserving the geometrical features of the cutting insert. The following parameters were used in cutting tests: feed f=0,1 mm/rev, depth of cut a_p=1 mm, cutting speeds v_c=315; 400 m/min.

ANALYSIS OF EXPERIMENTAL RESULTS

Roughness of the coatings defined by R_a parameter is within 0,60-0,64 μm range and is significantly higher than in case of the uncoated material surfaces. The R_a roughness parameter value ranges are 0,21-0,22 μm and 0,35-0,36 μm for cermets and cemented carbides respectively.
The surface roughness increase resulting from deposition of the TiN/(Ti,Al,Si)N/TiN gradient coatings should be attributed to the PVD process character – CAE and occurrence of the characteristic micro-particles, due to deposition of the pure titanium droplets, coming from the sputtered disk, and pits developing due to the titanium micro-particles dropping out immediately after completion of the coating deposition

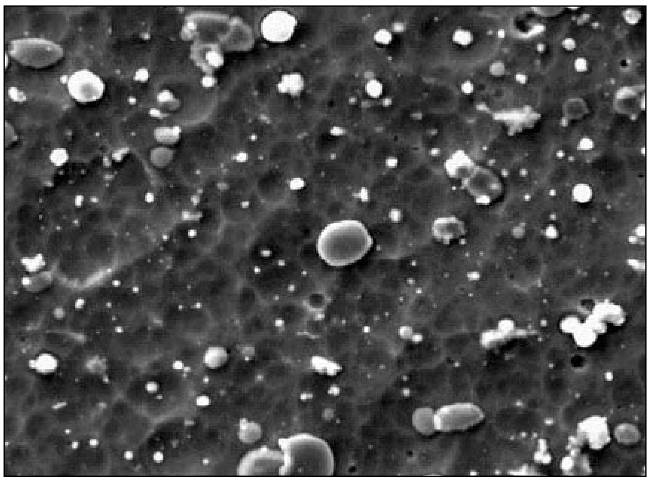

Fig. 1 – Topography of the TiN/(Ti,Al,Si)N/TiN gradient coating surface, deposited on the CC2 type cemented carbide substrate.

Fig.1 – Topografia del rivestimento superficiale TiN/(Ti,Al,Si)N/TiN di durezza elevata, depositato su substrato di carburi agglomerati del tipo CC2.

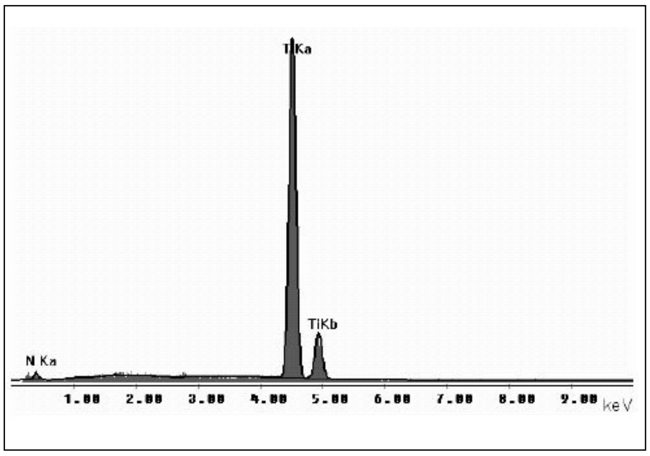

Fig. 2 – Plot of the X-ray dispersive energy spectrometer measurement from the droplet surface developed on the TiN/(Ti,Al,Si)N/TiN.

Fig. 2 – Tracciato del rilievo spettrometrico a raggi X per dispersione di energia della superficie "a gocce" presente sul rivestimento TiN/(Ti,Al,Si)N/TiN.

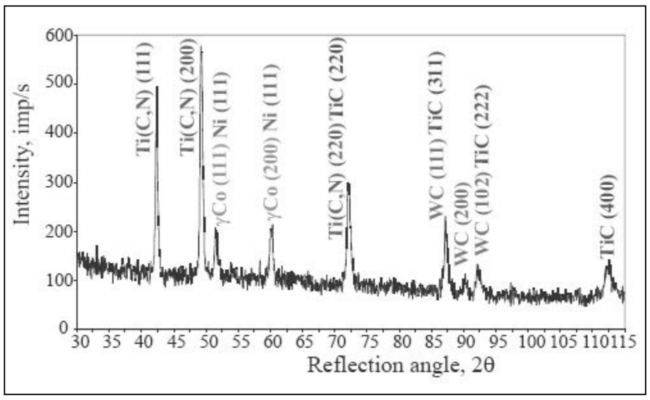

Fig. 3 – X-Ray diffractions pattern of the CER2 type cermet with the TiN/(Ti,Al,Si)N/TiN gradient coating.

Fig. 3 – Tracciato del diffrattogramma a raggi X del cermet tipo CER2 con rivestimento TiN/(Ti,Al,Si)N/TiN a gradiente di composizione.

process. Therefore, the surfaces of coatings demonstrate inhomogeneities connected with occurrences of the droplet shaped and elongated micro-particles, originating probably by their spattering when they hit the substrate surface during coating deposition process (Fig. 1). Examinations of the chemical compositions of the droplet shaped micro-particles made using the X-ray energy dispersive spectrometer (EDS) indicate that titanium
dominates inside of these micro-particles, which suggests that they are the pure titanium droplets knocked out from the titanium disk, which settle and solidify on the substrate surface (Fig. 2).
It was demonstrated, using the X-ray qualitative phase analysis methods, that – according to the initial assumptions - coatings containing the TiN type phases, and most probably the complex (Ti,Al,Si)N nitride one, were developed on surfaces of the investigated cemented carbides and cermets (Fig. 3).
Differentiation of the TiN and (Ti,Al,Si)N phases using the diffraction methods is impossible due to their isomorphous nature, as (Ti,Al,Si)N is – in fact – the secondary solid solution based on titanium nitride TiN.
Examinations of thin foils from gradient coatings confirm that, according to the original assumptions, coatings containing the TiN type phases were deposited onto the cemented carbide and cermet substrates. It is not feasible to differentiate these phases from the diffraction point of view, due to isomorphism of the TiN and (Ti,Al,Si)N phases (Figs. 4, 5).
It was found out, basing on the metallographic examinations of fractures made on the scanning electron microscope, that the TiN/(Ti,Al,Si)N/TiN deposited onto the investigated cer-

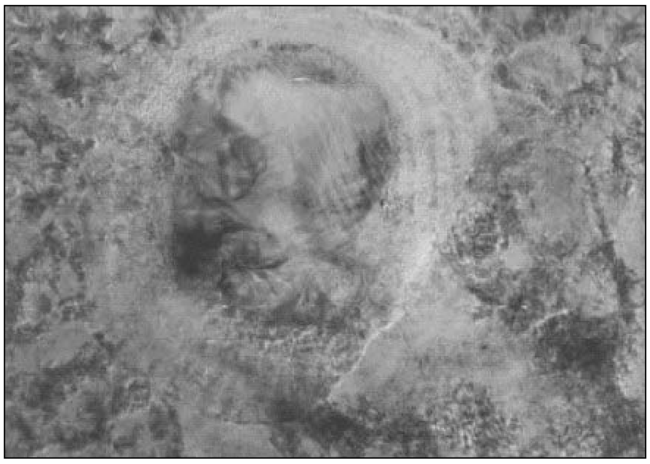

Fig. 4 – Thin foil structure from the TiN/(Ti,Al,Si)N/TiN gradient coating deposited on the CC2 cemented carbide.

Fig. 4 – Struttura presente su lamina sottile ottenuta da rivestimento di TiN/(Ti,Al,Si)N/TiN a gradiente di composizione, depositato su carburi sinterizzati del tipo CC2.

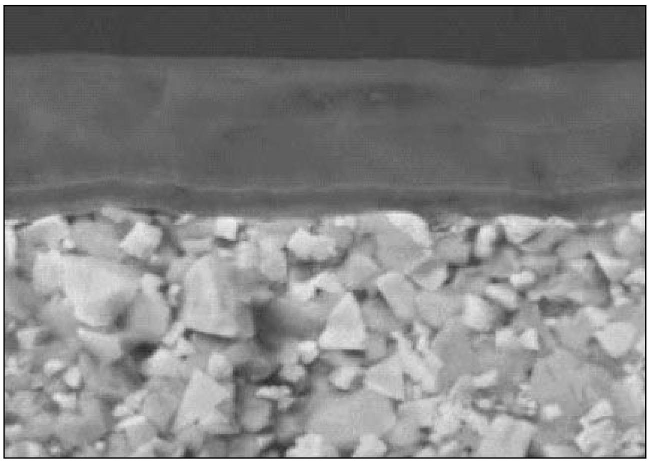

Fig. 6 – Fracture surface of the TiN/(Ti,Al,Si)N/TiN coating deposited onto the CC2 type cemented carbide substrate.

Fig. 6 – Superficie di frattura del rivestimento di TiN/(Ti,Al,Si)N/TiN depositato sul substrato di carburi agglomerati del tipo CC2.

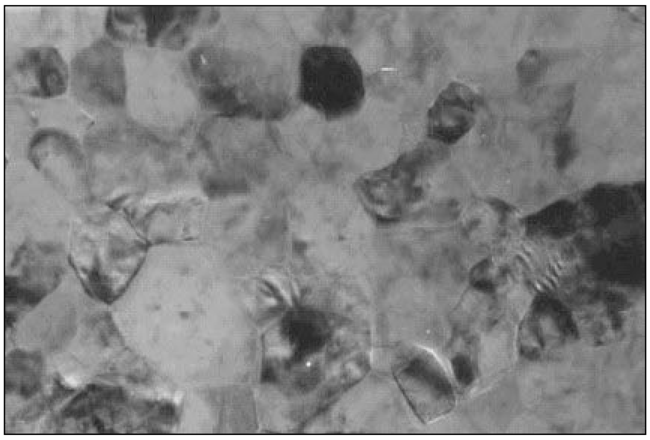

Fig. 5 – Thin foil structure from the TiN/(Ti,Al,Si)N/TiN gradient coating deposited on the CER2 cermet.

Fig. 5 - Struttura presente su lamina sottile ottenuta da rivestimento di TiN/(Ti,Al,Si)N/TiN a gradiente di composizione, depositato sul cermet tipo CER2 .

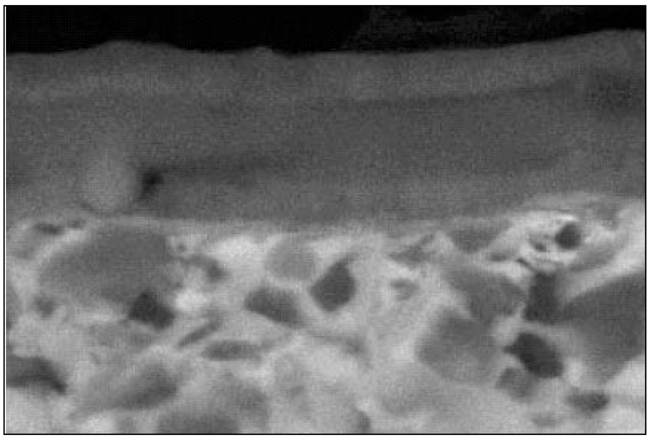

Fig. 7 – Fracture surface of the TiN/(Ti,Al,Si)N/TiN gradient coating deposited onto the CER2 type cermet substrate.

Fig. 7 - Superficie di frattura del rivestimento gradiente di TiN/(Ti,Al,Si)N/TiN depositato sul substrato di tipo cermet CER2.

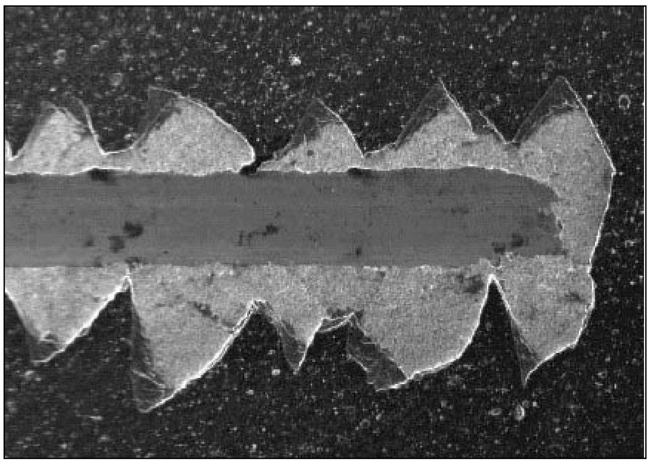

Fig. 8 – Indenter trace with the maximum load on the TiN/(Ti,Al,Si)N/TiN gradient coating surface deposited onto the CER2 cermet.

Fig. 8 – Traccia del penetratore con carico massimo sulla superficie di rivestimento gradiente di TiN/(Ti,Al,Si)N/TiN depositato sul cermet CER2.

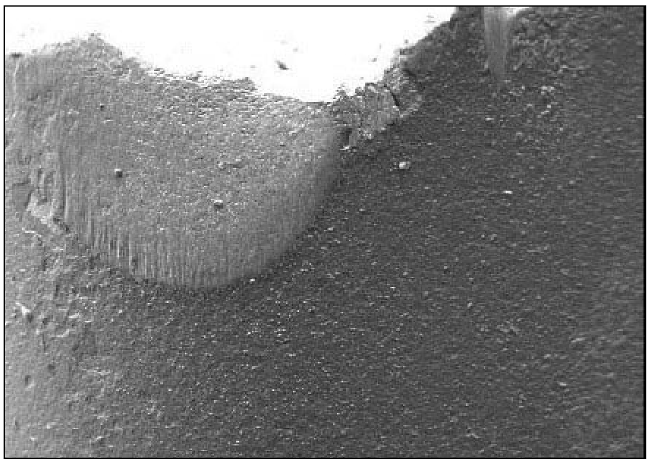

Fig. 10 – Flank wear nature of the CER2 type cermet with the TiN/(Ti,Al,Si)N/TiN coating (f=0.1 mm/rev, a_p=1 mm, v_c= 400 m/min).

Fig. 10 – Usura di natura laterale del cermet CER2 con il rivestimento TiN/(Ti,Al,Si)N/TiN (f=0.1 mm/rev, a_p=1 mm, v_c= 400 m/min).

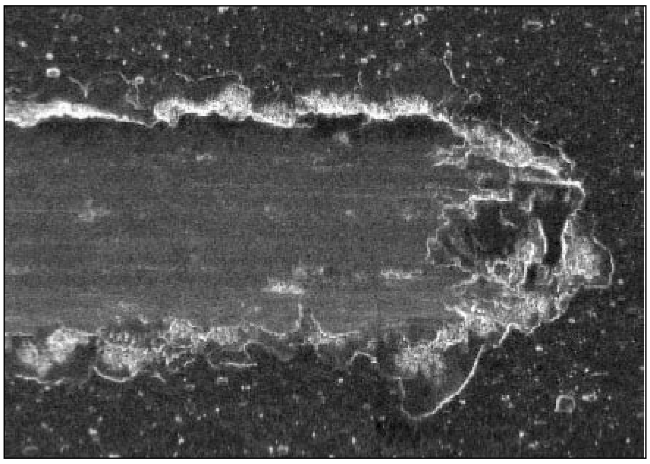

Fig. 9 – Indenter trace with the maximum load on the TiN/(Ti,Al,Si)N/TiN gradient coating surface deposited onto the CC2 cemented carbides.

Fig. 9 – Traccia del penetratore con carico massimo sulla superficie di rivestimento gradiente di TiN/(Ti,Al,Si)N/TiN depositato sui carburi agglomerati CC2.

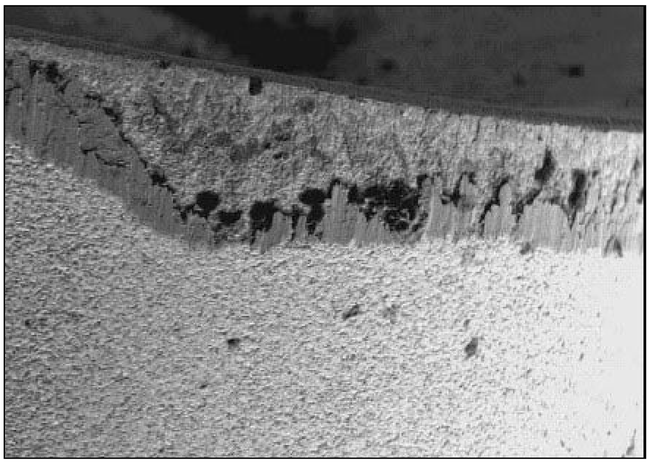

Fig. 11 – Flank wear nature of the CC1 type uncoated cemented carbides (f=0.1 mm/rev, a_p=1 mm, v_c= 315 m/min).

Fig. 11 - Usura di natura laterale dei carburi agglomerati del tipo CC1 non rivestiti (f=0.1 mm/rev, a_p=1 mm, v_c= 315 m/min).

mets and cemented carbides have the laminar packing. The particular TiN, (Ti,Al,Si)N gradient layers and the TiN interlayer have the poreless structure and adhere closely to each other, and the entire multi-layer coating adheres closely to the substrate (Figs. 6, 7). Basing on chemical composition analysis of the investigated materials' fractures made using the X-ray energy dispersive spectrometer (EDS) it was found out that according to the initial assumptions coatings containing titanium, aluminum, silicon, and nitrogen, constituting gradient coatings of the TiN/(Ti,Al,Si)N/TiN types, were deposited on the substrates from cemented carbides and cermets.

The TiN/(Ti,Al,Si)N/TiN gradient coatings are characterized by a good adhesion to substrate from the cemented carbides and a significantly better adhesion to cermets. The critical load L_c (AE) values for the gradient coatings deposited onto the cermet and cemented carbide substrates is 131,2 N and 77,1 N respectively. The significantly better adhesion of the coatings to the cermet substrate than to the cemented carbide one may result from crystallization of the layers with plasma in the CAE method. In case of depositing the coatings onto the cermets, the source of the nitrogen for the developing coating is not only the working gas, but also nitrogen coming from the substrate alone, making diffusion mixing of elements in the interlayer easier. Therefore, not only the adhesion decides the adherence, but also the diffusion mixing of elements in the interlayer, between the substrate and the coating, with two simultaneously available sources of diffusion of elements constituting the coatings and titanium from the coating to the substrate, and also of nitrogen, and perhaps also titanium from the substrate to the coating, the more so, as the substrate temperature during the process is 550°C.

Failures of the TiN/(Ti,Al,Si)N/TiN gradient coatings deposited onto CC2 type cemented carbides are characterized by multiple two-sided coating chipping on the crevice edges and scratches and delamination inside thecrevice, leading to local delamination of the coating at its ending section where it contacts the crevice. Increasing the load during the test leads to intensification of chipping and joining the craters in the "tooth-like" form at the crevice edges, which leads to the band-form, partial coating delamination. In case of the TiN/(Ti,Al,Si)N/TiN gradient coatings deposited on the CER2 type cermets another failure type was observed. The

Designation	Coating Type	Coating Composition	Microhardness $HV_{0,07}$	Tool life T, min	Roughness, R_a, µm
CC1			1900	4	0,35
CER1			2450	17	0,22
CC3	monolayer	TiN	2000	8	0,59
CC2			3190	27	0,64
CER2	gradient layer	TiN/(Ti,Al,Si)N/TiN	3310	55	0,60
CC4	multilayer	TiCN/Al2O3 /TiN	2300	23	0,60
CER3		TiN/TiC/TiN	3000	35	0,79

Table 3 – Comparison of properties of the investigated cemented carbides and cermets.

Tab. 3 – Confronto delle proprietà dei carburi agglomerati e cermet esaminati.

first coating failure symptoms are the conformal cracks resulting from tension, turning into single spallings located at the bottom of the developing crevice and in the coating-crevice contact zone. Chipping and spalling failures develop in the central zones of the crevices and at their edges in the form of the fine arc-shaped craters.
Similar effects can be observed at the edges in the ending part of the crevice. Semicircles connected with the conformal cracking occur at the crevice bottom at the big load force, attesting the fragmentation, local delamination, and consequent relocation of the torn coating fragments along with the plastic strain of the substrate by the traveling indenter. In all examined cases, even at the biggest loads, total delamination never occurs for any of the investigated coatings (Fig. 8, 9).
Microhardness of the investigated uncoated CER1 cermet is 2450 $HV_{0,07}$, and for the CC1 cemented carbide it is 1900 $HV_{0,07}$. Depositing the TiN/TiAlSiN/TiN gradient coating on such substrate results in a significant increase of the surface layer hardness, in the range of 3190- 3310 $HV_{0,07}$ (Table 3). Therefore, depositing the wear resistant coatings onto the tool cermet and cemented carbide substrates results in a significant increase of the surface layer microhardness, contributing in this way in machining to the decrease of the wear intensity of cutting tools' flanks made from cemented carbides and from cermets.
To verify correlation between hardness of the investigated materials and working properties of the multipoint inserts, single point turning tests were made. All work tests have the comparative character, i.e., life of inserts was determined basing on the wear land width measurements on the tool flank after machining, at the same turning parameteres, for a predeterminedd period of time (Table 3).
Depositing the wear resistance TiN/(Ti,Al,Si)N/TiN gradient coatings on cemented carbides and cermets results in increase of their wear resistance, which immediately causes, among others, increasing the tool flank life.
Therefore, depositing coatings on the investigated materials and connected with it a significant surface layer microhardness increase reduces wear rate of tool flanks made from these tool materials. In case of inserts with the TiN/(Ti,Al,Si)N/TiN coating, the longest tool life has been demonstrated by the CER2 type cermet, for which the VB= 0,2 mm tool flank wear land width criterion value was exceeded after 55 minutes of the experiment (Fig. 10), and the shortest tool life has the CC2 type cemented carbide for which the tool flank wear land width criterion value was exceeded after 27 minutes.
Comparison tests of the uncoated inserts from cermets and cemented carbides carried out in the same cutting conditions demonstrate the accelerated, and in many cases catastrophic failures of the cutting tool flanks. Cermet of the CER1 type, for which the VB= 0,2 mm tool flank wear land width criterion value was exceeded after 17 minutes of the test duration, has the longest tool life, whereas the shortest tool life is characteristic for the CC1 type cemented carbide, for which the same tool flank wear width criterion was exceeded after 4 minutes of machining (Fig. 11).

CONCLUSIONS

Gradient coatings of the TiN/(Ti,Al,Si)N/TiN systems deposited with the PVD method in the CAE process onto the substrates of cermets and cemented carbides demonstrate better working properties than the commercially available tool materials with the single – and multiple-layer, as well as single– and double-component coatings deposited both in the PVD and CVD processes.
Cutting tests confirm then the advantages of the TiN/(Ti,Al,Si)N/TiN type coatings obtained with the PVD method in the cathodic arc evaporation process on the cemented carbides and tool cermets, as a material significantly reducing abrasive, thermal and adhesion wear, which directly influences, among others, extension of tool life, in comparison wear to the commercially available uncoated tools and with the single- and multiple-layer coating deposited with the PVD/CVD methods.
The TiN/(Ti,Al,Si)N/TiN system coatings deposited with the PVD method in the Cathode Arc Evaporation onto the substrates from cermets and sintered carbides reveal better working properties in comparison to the commercial tool materials with the mono- or multi-layer and single and two-component coatings deposited in the PVD or CVD processes, and therefore, and also because of the possibility of employing them in the pro-ecological dry cutting processes without any cutting fluids they qualify for multiple industrial applications for cutting tools.

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ABSTRACT

CARATTERIZZAZIONE DI RIVESTIMENTI DI TiN/(Ti,Al,Si)N/TiN DI DUREZZA ELEVATA DEPOSITATI SU MATERIALI SINTERIZZATI PER UTENSILI

Parole chiave: trattamenti superficiali, rivestimenti, tecnologie

Questo lavoro presenta i risultati di analisi strutturali, di accertamenti delle proprietà meccaniche e in esercizio di rivestimenti sottili di TiN/(Ti,Al,Si)N/TiN a gradiente di composizione, resistenti all' usura, depositati con processo Cathode Arc Evaporation - CAE, su substrato sia di cermet che di carburi sinterizzati. Le analisi strutturali sono relative ai rivestimenti applicati e al loro substrato e si sono avvalse di osservazioni con microscopia elettronica (SEM e TEM), microscopia ottica, diffrazione a raggi X.

La valutazione dell'adesione dei rivestimenti depositati sui cermet e sui carburi sinterizzati è stata effettuata utilizzando la prova di graffio. Le proprietà di resistenza al taglio dei materiali studiati sono state determinate sulla base delle prove di taglio continuo dell'acciaio C45E. Sono stati condotte misure di durezza del substrato e di microdurezza dei rivestimenti depositati , mediante misuratore di ultra-microdurezza con un carico di 70 mN. Inoltre è stata rilevata la rugosità superficiale prima della deposizione dei rivestimenti e dopo completamento del processo di deposizione PVD.