Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 14
ISSUE 1-2
January-February
2006

Gradient coatings deposited by Cathodic Arc Evaporation: characteristic of structure and properties

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Received 15.11.2005; accepted in revised form 31.12.2005

Properties

ABSTRACT

Purpose: It has been demonstrated in the paper that deposition of the multilayer and gradient coatings with the PVD method in the Cathodic Arc Evaporation CAE process on tools made from cermets.

Design/methodology/approach: Structural examinations are presented of the applied coatings and their substrate made on the TEM, SEM and on the light microscope. Evaluation of the adhesion of the deposited coatings onto the 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. Microhardness tests of the deposited coatings were made on the ultra-micro-hardness tester. Surface roughness tests were also made before depositing the coatings and after completing the PVD process.

Findings: It has been demonstrated in this work that deposition of the TiN+(Ti,Al,Si)N+TiN multilayer or gradient coatings with the PVD (Cathodic Arc Evaporation) process on tools made from cemented carbides, results in the increase of coatings' hardness and improvement of their adhesion to the substrate, in comparison with the multiple-layer coatings deposited using the PVD method on the same substrate materials, deciding improvement of the working properties of cutting tools coated with the TiN+gradient or multi(Ti,Al,Si)N+TiN system coatings, compared with coatings developed on the same sintered tool materials, but uncoated or coated with simple coatings.

Practical implications: Gradient coatings deposited with the CAE method onto the cermet substrate qualify for the widespread industrial use on cutting tools and offering the possibility to use them in the pro-ecological dry cutting processes without the use of the cutting fluids and in the „Near-Net-Shape" technology.

Originality/value: In the paper the research of newly worked out multilayer and gradient TiN+(Ti,Al,Si)N+TiN nanocrystalline coatings deposited in the PVD method on cermets carried out in order to improve the tool cutting properties. To explain reasons of significant improvement of tool exploitation properties modern material science methods of research especially of the one of thin foils including those on cross-sections on a high-voltage transmission electron microscopy have been used. As a result of very complicated and difficult preparation methods, similar research are made only by few, the most advanced in the world, laboratories of electron microscopy, but concerning other coatings on other substrates than those analysed in the given paper. Undoubtedly, using such modern research methods is a modern approach to the solution and explanation of the scientific problem presented in that paper.

Keywords: Gradient coatings; Multilayer coatings; Cermets; PVD; TEM

1. Introduction

Tool cermets have been widely applied to high speed finishing and machining of difficult-to-cut materials. High-quality tool cermets are competing with uncoated and coated carbides in the field of machining. Despite the high wear resistance of cermets and their low adhesive tendency, coatings also offer advantages to these cutting materials.

Deposition of hard anti-wear coatings based on transition metal carbides, nitrides, or oxides onto the sintered tool materials (including cermets) features one of the most intensely developing research directions, stimulated by the growing working requirements posed to machines and equipment, making it possible to improve significantly their properties. Using tools with such coatings reveals the significant increase of their life, compared with the conventional ones. The cathodic arc evaporation is characterized especially for their high degree of ionization of the vaporized atoms, which resulted in effective ion bombardment of substrates.

Cermets coated with the wear resistance layers belong to the contemporary sintered tool materials with a fast growing importance in machining technology. Broader and broader employment of cermets – the fastest developing tool materials – is connected with transition of machining of semi-products from roughing to semi-finishing or finishing in one setting. The contemporary tool material like cermet meets in many cases requirements of new technologies, and first of all of the *„Near-Net-Shape”* one [1-12].

The goal of this work has been examining the influence of deposition in the CAE process the coatings of the TiN+gradient or multi (Ti,Al,Si)N+TiN types on structure and properties of cermets, and comparing them with the commercial tool cermets.

2. Examination procedure

2.1. Substrate

The cermets used as for the substrate in the experiments are a commercial inserts for turning application. The SNMG 120408 type (designation according to ISO 1832:1991) inserts were used in the experiments. Chemical composition of the investigated materials are given in Table 1.

2.2. Coating

The investigations were carried out on tool cermets: uncoated and coated using the PVD method in the (CAE - Cathodic Arc Evaporation) process of Physical Vapour Deposition of coatings from the gaseous phase, with the TiN+gradient or multi (Ti,Al,Si)N+TiN wear resistant coatings. Conditions of coating deposition: substrate polarisation -200 V, substrate temperature 550°C, pressure in the chamber 0,2 Pa, working atmosphere N_2.

2.3. Structure and properties – examination procedure

The examinations of thin foils were made on the JEOL 3010CX transmission electron microscopy 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.

Observations of surfaces and structures of the developed coatings were carried out on the transverse fractures on the scanning electron microscope (SEM). To obtain the fracture images 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.

Tests of the coatings’ adhesion to the substrate material were made using the scratch test, routinely employed in case of the coatings obtained in the PVD processes. The tests were made on the CSEM REVETEST device. The examinations were carried out 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 micro-hardness tests were made on the SHIMADZU DUH 202 ultra micro-hardness tester. The tests were made at the 70 mN load.

Table 1
Chemical composition of the investigated cermets

Designation	Mass concentration of elements, %						
	C	N	Ti	Ta	Ni	Co	W
C1, C3, C5, C7	0.80	2.00	48.70	13.50	4.70	8.60	21.65
C2, C4, C6	1.65	2.40	47.50	10.70	8.80	8.00	20.95

Table 2
Specification of the tested cermets

Designation	Coating	Coating thickness, μm	Process type
C1	uncoated	–	–
C2	uncoated	–	–
C3	TiN+gradient (Ti,Al,Si)N+TiN	4.0	PVD (CAE)
C4	TiN+gradient (Ti,Al,Si)N+TiN	4.0	PVD (CAE)
C5	TiN+multi (Ti,Al,Si)N+TiN	4.0	PVD (CAE)
C6	TiN+multi (Ti,Al,Si)N+TiN	4.0	PVD (CAE)
C7	TiN+TiC+TiN (commercial)	5.0	PVD

The roughness measurements of the developed coatings and the substrate were made in two orthogonal directions on the Taylor-Hobson Sutronic3+ device. The R_a parameter was assumed to be the value describing the surface roughness.

2.4. Machining test

Working properties of the developed coatings were determined basing on the technological cutting tests of the continuous dry turning of the C45E type (according to PN-EN 10083+A1:1999) unalloyed steel. Inserts' life was determined basing on the wear land measurements on the tool flank, measuring the average wear land VB and the maximum one VB_{max} after cutting for a predetermined period. Cutting tests were stopped when the VB value exceeded the assumed criterion for finishing, i.e., VB= 0,2 mm., specifying the tool life T in minutes. 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=250; 315; 400 m/min.

3. Discussion of the test results

Examinations of thin foils from coatings confirm that, according to the original assumptions, coatings containing the TiN type phases were deposited onto the cermets 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. Structures of coatings deposited onto cermets is presented in Figure 1.

It was found out, basing on the metallographic examinations of fractures made on the scanning electron microscope, that the TiN+gradient or multi (Ti,Al,Si)N+TiN coatings deposited onto the investigated tool cermets have the laminar packing. The particular TiN, gradient or multi (Ti,Al,Si)N layers and the TiN interlayer have the poreless structure and adhere closely to the substrate with no cracks or discontinuities (Figs.2,3). Observations of the surface morphology reveal its inhomogeneity connected with occurrences of numeorus drop-shaped micro-particles on them (Fig.4). 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.5). Basing on the surface morphology observations on the scanning electron microscope of the commercial tool cermets, in case of coatings put down with the PVD method (on C7 cermet), the inhomogeneity was revealed connected with the occurrences of

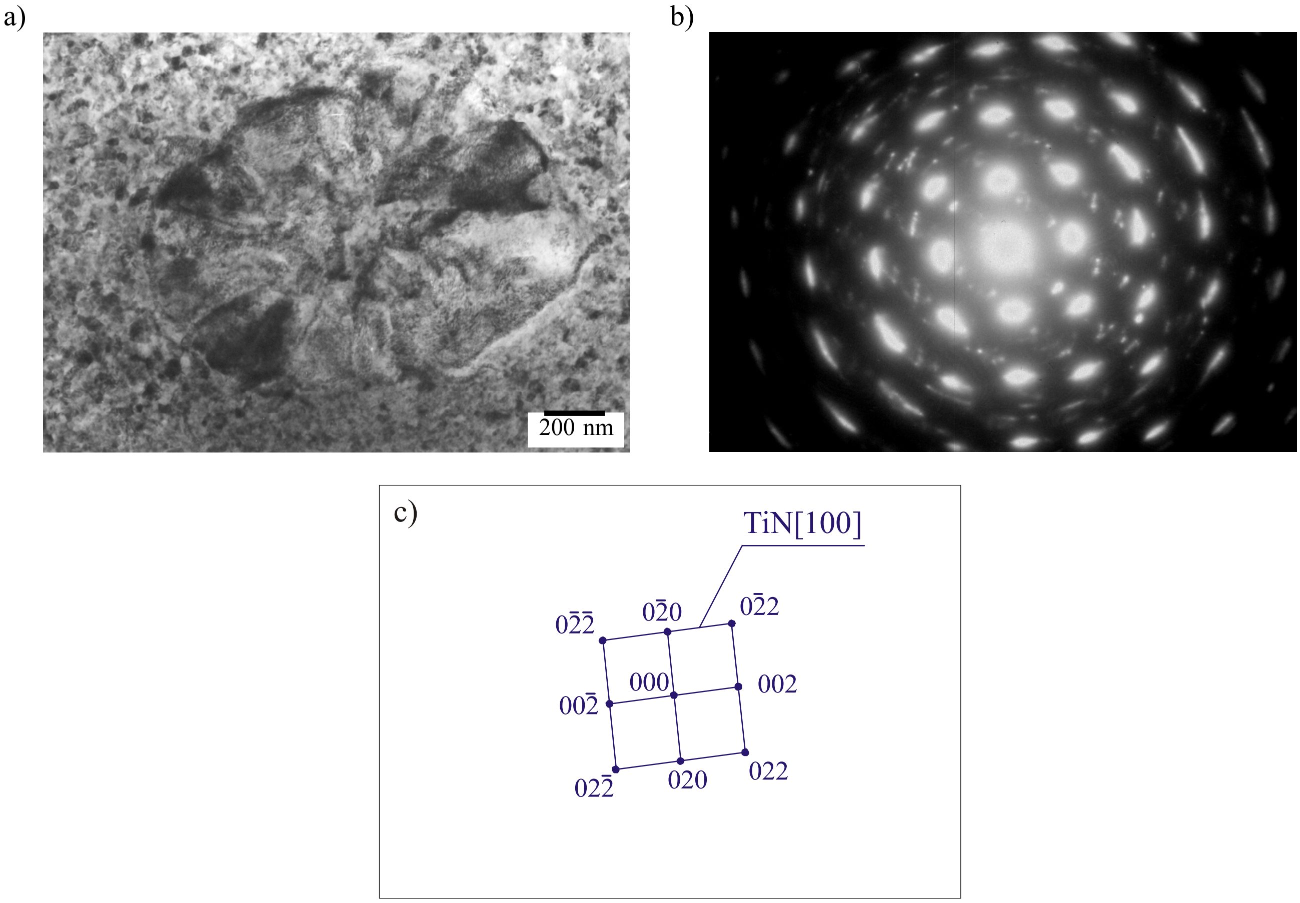

Fig. 1. Structure of the thin foil from the TiN+gradient (Ti,Al,Si)N+TiN coating deposited onto the C3 type cermet, b) diffraction pattern from the area as in figure a, c) solution of the diffraction pattern from figure b.

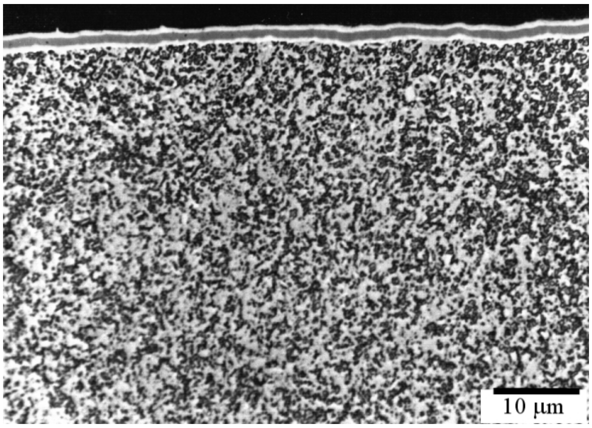

Fig. 2. Skew microsection C5 type cermet with the TiN+gradient (Ti,Al,Si)N+TiN coating, magnification 500x

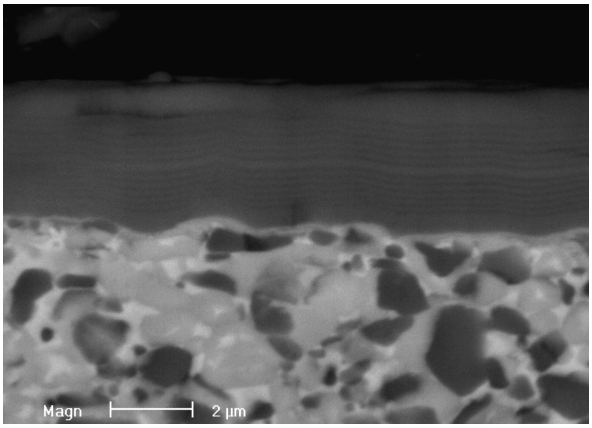

Fig. 3. Fracture surface of the TiN+multi (Ti,Al,Si)N+TiN coating deposited onto the C3 type cermet substrate

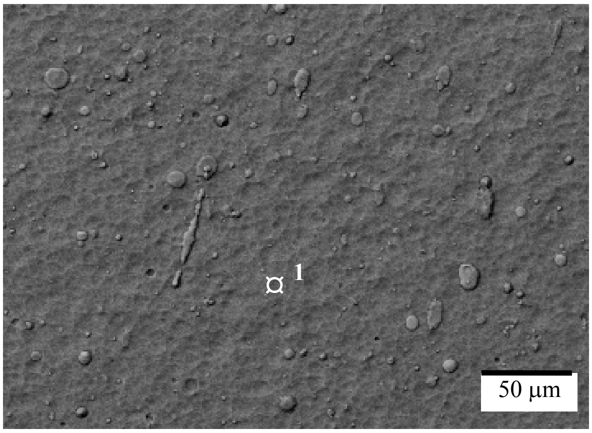

Fig. 4. Topography of the TiN+multi (Ti,Al,Si)N+TiN coating surface, deposited in the PVD processes on the C3 type cermet substrate

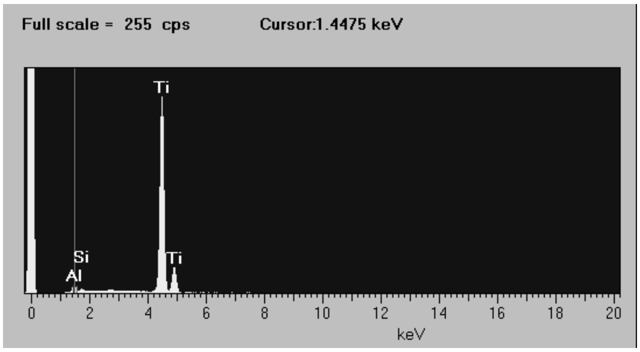

Fig. 5. Plot of the X-ray dispersive energy spectrometer measurement from the surface (point 1) developed on the TiN+multi (Ti,Al,Si)N+TiN coating

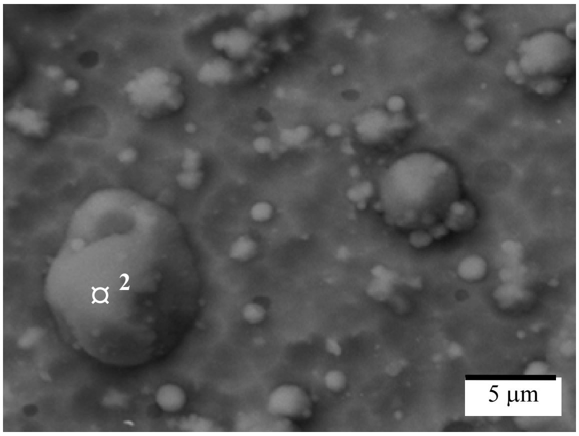

Fig. 6. Topography of the TiN+TiC+TiN commercial coating surface, deposited in the PVD processes on the C7 type cermet substrate

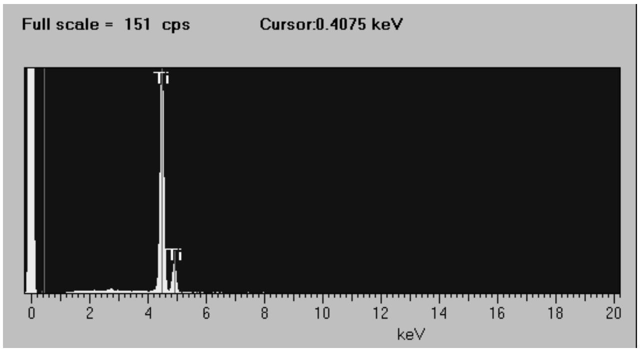

Fig. 7. Plot of the X-ray dispersive energy spectrometer measurement from the droplet (point 2) surface developed on the TiN+TiC+TiN commercial coating

many drop shaped and elongated micro-particles on surface (Fig.6). The sizes of the drop-shaped micro-particles are differentiated and vary from several tenths of a micrometer to above 10 μm. Examinations of chemical composition of the micro-particles using the X-ray energy dispersive spectrograph EDS indicate that titanium prevails inside of the micro-particles, which suggests that these are the molten metal drops solidifying on the substrate surface (Fig.7).

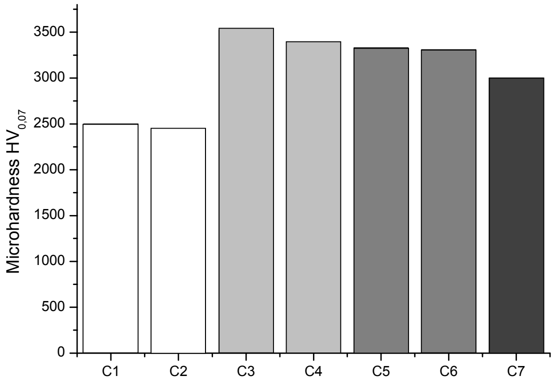

Fig. 8. Hardness values of coatings deposited onto the tool cermets

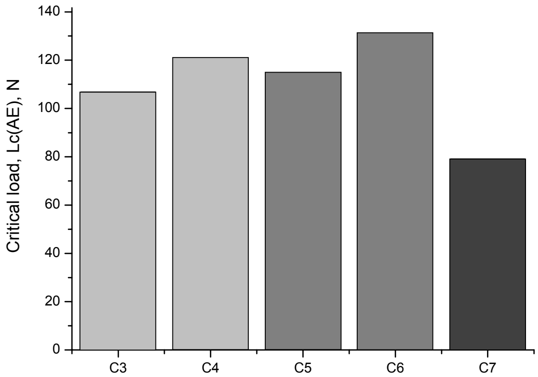

Fig. 9. Critical loads Lc (AE) determined for coatings deposited onto the tool cermets

Basing on the roughness tests surface profiles were determined for the deposited coatings and average deviations of the R_a roughness profile was determined. It was found out that the smallest value of R_a=0,2 μm is characteristic for the C1 specimen surface, whereas the surface of the C2 type cermet displays R_a=0,22 μm. After depositing the TiN+gradient (Ti,Al,Si)N +TiN coatings onto the cermet substrate, the surface layer roughness increases and is within the range of R_a=0,60-0,67 μm.

The surface roughness increase after depositing the coatings should be – probably – linked with the character of the CAE process, which was confirmed with examinations of the surface morphology on the scanning electron microscope.

Basing on the micro-hardness tests it was found out that the micro-hardness of the uncoated C2 and C1 cermets was 2450 and 2500 $HV_{0,07}$ respectively (Fig.8). Depositing the TiN+gardient (Ti,Al,Si)N+TiN coating onto the cermet substrate results in the significant surface hardness increase within the range of 3310-3330 $HV_{0,07}$. In case of the TiN+multi(Ti,Al,Si)N+TiN coatings a significant increase of the surface layer was also observed. Hardness of the TiN+multi(Ti,Al,Si)N+TiN coating is 3390-3520 $HV_{0,07}$. No relationship was found between the substrate hardness and hardness of the deposited surface layer.

The critical load values L_c (AE) were determined using the scratch method with the increasing load, characterizing adhesion of the investigated coatings to the cermet substrate (Figs.9, 10). It was found out in case of the TiN+gradient (Ti,Al,Si)N+TiN coating that the critical load values of L_c=131,2 N and L_c=114,8 N are displayed by a coatings put down on tool cermet, for the C4 and C3 types respectively. Similar relationships were observed in case of the TiN+multi(Ti,Al,Si)N+TiN coating. For the C6 type cermet the value of L_c=120,9 N was found out, and for the C5 type cermet L_c=114,8 N was obtained. In case of the commercial coatings deposited onto the C7 cermet value of L_c=57,27 N was obtained.

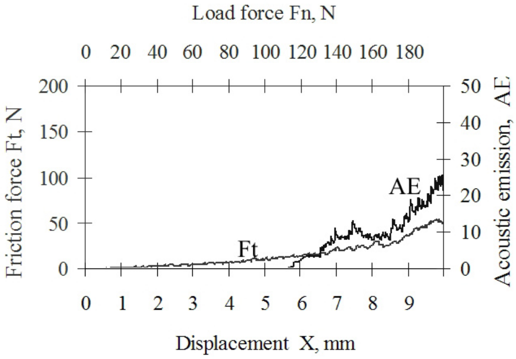

Fig. 10. Plot of the dependence of the acoustic emission AE and friction force Ft on the load force for the C4 cermet with the TiN+gradient (Ti,Al,Si)N+TiN coating

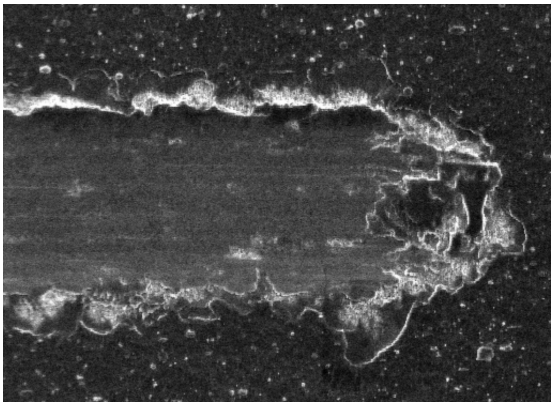

Fig. 11. Indenter trace with the maximum load on the TiN+gradient (Ti,Al,Si)N+TiN coating surface deposited onto C4 tool cermet

In case of the TiN+gradient or multi (Ti,Al,Si)N+TiN coatings deposited on the cermets it was observed that the 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 are observed at the edges in the ending part of the crevice. Single failures are often connected forming bands of the local coating delamination, not more. In all examined cases, even at the biggest loads, total delamination never occurs for any of the investigated coatings (Fig.11).

It was found out, basing on the turning tests carried out with the tool cermets, that a clear anti-wear effect of the TiN+gradient or multi (Ti,Al,Si)N+TiN coatings on the cutting inserts' life was demonstrated. In case of cutting inserts with the TiN+multi (Ti,Al,Si)N+TiN coating put down, for the following turning parameters: cutting speed v_c = 400 m/min, feed f = 0,1 mm/rev, depth of cut a_p = 1,0 mm, it was observed that the longest tool life was obtained for the C6 type cermet, for which the VB= 0,2 mm tool flank wear land width criterion values were exceeded after 60 minutes of continuous turning, and for the C5 type cermet this criterion was reached after 43 minutes. A similar ranking was obtained in case of cutting inserts with the TiN+gradient (Ti,Al,Si)N+TiN coating put down. For the following cutting parameters: cutting speed v_c = 400 m/min, feed f = 0,1 mm/rev, depth of cut a_p = 1,0 mm, it was observed that that the longest tool life was obtained for the C4 type cermet, for which the VB= 0,2 mm tool flank wear land width criterion values were exceeded after 55 minutes of the experiment duration, whereas for the C3 type cermet after 43 minutes. The comparative tests of the uncoated cermet inserts carried out in the same cutting conditions revealed that the longest life was obtained for the C2 type cermet, for which the VB= 0,2 mm tool flank wear land width criterion value was exceeded after 17 minutes of continuous turning, whereas for the C1 type cermet the tool flank wear land width criterion value was exceeded after 14 minutes of cutting at the cutting speed of v_c = 400 m/min. Comparative tests of the commercial multi-point inserts made from the tool cermets coated in the PVD process, carried out in the same cutting conditions revealed that the longest tool life was for the C7 type cermet, for which the VB= 0,2 mm tool flank wear land width criterion value was exceeded after 35 minutes of continuous turning.

4. Conclusions

It was found out, basing on the metallographic examinations that the TiN+gradient or multi (Ti,Al,Si)N+TiN coatings deposited onto the tool cermets in the cathodic arc evaporation (CAE) have the laminar packing, poreless structure and adhere closely to each other, and the entire coatings have very good adhesion to the cermet substrate. In all investigated cases of adhesion, total delamination never occurred for any of the investigated coatings, even at the highest load. The technological turning tests of cutting ability of the C45E steel reveal that the cermet multi-point inserts with the TiN+gradient or multi (Ti,Al,Si)N+TiN coatings put down, are characterized by the significantly better wear resistance compared to the uncoated commercial multi-point inserts and with the commercial coatings of the TiN+TiC+TiN type. The TiN+gradient or multi (Ti,Al,Si)N+TiN system coatings deposited with the CAE method onto the cermet substrate qualify for the widespread industrial use on cutting tools and offering the possibility to use them in the pro-ecological dry cutting processes without the use of the cutting fluids and in the *„Near-Net-Shape”* technology.

Acknowledgements

Researches were financed partially within the framework of the Polish State Committee for Scientific Research Project PBZ-100/4/2004 headed by Prof. L.A. Dobrzański.

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