Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 19
ISSUE 2
December
2006

Improvement of tool materials by deposition of gradient and multilayers coatings

L.A. Dobrzański*, K. Gołombek, J. Mikuła, D. Pakuła
Division of Materials Processing Technology, Management and Computer Techniques in Materials Science, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland
* Corresponding author: E-mail address: leszek.dobrzanski@polsl.pl

Received in revised form 25.10.2006; accepted 31.10.2006

Manufacturing and processing

ABSTRACT

Purpose: Investigation of the functional properties of cermets, Si_3N_4 and Al_2O_3 based ceramics, coated with the PVD and CVD multilayer and gradient coatings and comparison them with the commercial uncoated and coated tool materials.

Design/methodology/approach: TEM, SEM, confocal microscopy, scratch test, microhardness tests, roughness tests, cutting tests.

Findings: Employment of the hard wear resistant coatings deposited onto the sintered ceramic tool materials with the physical deposition from the gaseous phase (PVD) is reckoned as one of the most important achievements in the last years in the area of improvement of the service properties of ceramic cutting tools. Depositing the anti-wear coatings of the gradient and multi TiN+(Ti,Al,Si)N+TiN types onto the investigated ceramic tool materials makes it possible to achieve the clear improvement of their tool life and also of the quality of the machined surfaces, reduction of machining costs and elimination of cutting fluids used in machining.

Practical implications: The widespread use in machining of oxide and nitride ceramics, as well as of cermets with the complex nanocrystaline coatings deposited in the PVD processes contributes to the increased interest in the contemporary „Near-Net-Shape" technology, i.e., manufacturing semi-products with the shape and dimensions as close as possible to those of the finished products.

Originality/value: In the paper the research of multilayer and gradient TiN+(Ti,Al,Si)N+TiN nanocrystalline coatings deposited in the PVD method on sintered tool materials carried out in order to improve the tool cutting properties.

Keywords: Thin & thick coatings; Tool materials; Cutting; PVD; CVD

1. 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) [1-4]. The causes of the cutting tools wear are as follows: mutual interactions - mechanical, thermal, and molecular of the edge with the machined material and formed chip, occurring in the contact zone. Several cutting tools wear have been developed, taking into account the qualitative and quantitative effects of their causes. Wear of the cutting tools edges takes place in the complex conditions caused, among others, by the mutual interactions of the mechanical- and fatigue wear, and plastic strain, as well as phenomena connected with the adhesion, thermal-, and diffusion wear and oxidation [5-14].

Moreover, each combination of the substrate material – coating type – deposition method calls for determining properties of the coated material and defining, basing on them, the range of its possible applications. Research in this area is concentrated among other on searching new composite gradient coatings, both multicomponent and multilayer ones, and adding of new elements to coating combinations used since many years, like silicon or vanadium to TiAlN [1-18].

The goal of this work is investigation of the structure and functional properties of cermets, Si_3N_4 and Al_2O_3 based ceramics, coated with the PVD and CVD multilayer and gradient coatings and comparison them with the commercial uncoated and coated tool materials.

2. Materials and methods

Experiments were carried out on the multi-point inserts made from the Si_3N_4 nitride ceramics, Al_2O_3+ZrO_2 oxide ceramics, cermets with the multi-layer and multi-component layers deposited in the PVD process with the (Ti,Al,Si)N type coatings, and in the CVD process with the Al_2O_3 and TiN coating combination, which were later compared with the commercial inserts (Table 1).

Table 1.
Characteristics of the PVD and CVD coatings deposited on the sintered tool materials

Substrate	Coating			Process type
	Type	Composition	Thickness,	
Si_3N_4	uncoated			
Al_2O_3+ZrO_2				
Cermet				
Si_3N_4	gradient layer	TiN+ (Ti,Al,Si)N+TiN	2.0	PVD
Al_2O_3+ZrO_2			2.0	
Cermet			4.0	
Si_3N_4	multi layer	TiN+multi (Ti,Al,Si)N+TiN	4.0	
Al_2O_3+ZrO_2			2.3	
Cermet			4.0	
Cermet		TiN+TiC+TiN[1)]	5.0	
Si_3N_4	two layer	TiN+Al_2O_3	10.0	CVD
Al_2O_3+ZrO_2			6.0	
Si_3N_4		Al_2O_3+TiN[1)]	2.6	

[1)] commercially available inserts from various manufacturers

The microhardness tests using the Vickers method were made on the Shimadzu DUH 202 tester.

Adhesion evaluation of the coatings on the investigated inserts was made using the scratch test on the CSEM REVETEST device.

Topography examinations of the tool surface after scratch test were carried out on the OLYMPUS LEXT OLS3000 confocal microscope.

The roughness measurements of the developed coatings and substrate were made Surtronic3+ device.

Analysis of the chemical composition as function of the distance from the specimen surface, the so called profile analysis, and changes of concentrations in the interlayer between the coating and the substrate material were evaluated basing on examinations made in the LECO Instruments GDOES-750A.

The examinations of thin foils were made on the JEOL 3010CX TEM at the accelerating voltage of 300 kV. The diffraction patterns from the TEM were solved using the computer program.

The metallographic examinations of the investigated materials (coatings and substrates) were made on the Philips XL-30 and JEOL JSM 5610 one equipped with X-ray energy dispersive spectrometer.

Cutting ability of the investigated materials was determined basing on the technological continuous cutting tests of the EN-GJL-250 grey cast iron and C45E steel. The VB = 0.20 (oxide ceramics, cermets) and 0.30 mm (nitride ceramics) width of the wear band on the surface of the tool used for machining was the criterion of the cutting edge consumption evaluation. The following parameters were used in the machining capability experiments: feed rate f = 0.10; 0.15; 0.20 mm/rev; depth of cut ap = 1; 2 mm; cutting speed vc = 200; 400m/min.

3. Results

The microhardness tests revealed that the uncoated nitride, oxide ceramics and cermet has hardness within 18.5-24.50 GPa range respectively. Deposition of the PVD and CVD coatings onto the specimens causes the surface layer hardness increase reaching from 19.20 to 40.90 GPa, that is up to 100% more compared to the substrate hardness (Fig. 1).

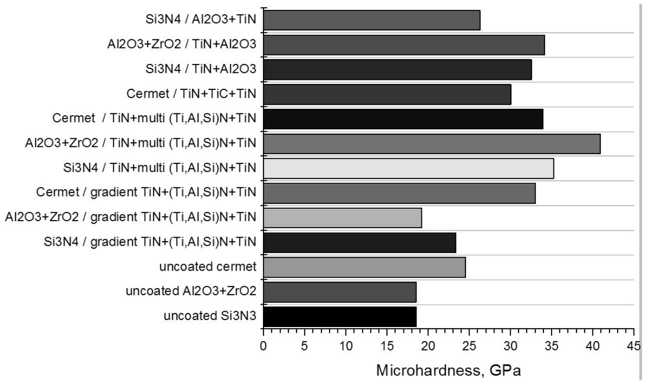


Fig. 1. Comparison of microhardness results

The critical load values L_c (AE) were determined using the scratch method with the linearly increasing load („scratch test"), characterising adherence of the investigated PVD/CVD coatings to the cermets and tool ceramics. The critical load was determined as the one corresponding to the acoustic emission increase signalling beginning of spalling of the coating. The coatings deposited onto the investigated substrates are characterised by good adherence (L_c = 40-137 N), only to the TiN+(Ti,Al,Si)N+TiN gradient coating deposited onto the nitride ceramic substrate has a lower adherence equal L_c=22 N (Fig. 2).

Defects of the investigation coatings, and especially of the three layer (Ti,Al,Si)N based ones, deposited onto the nitride ceramics are characterised by a significant number of the coating spalling defects on both scratch edges and by delamination within the scratch ending up at its final part with the local coating delamination at its contact with the scratch (Fig. 3).

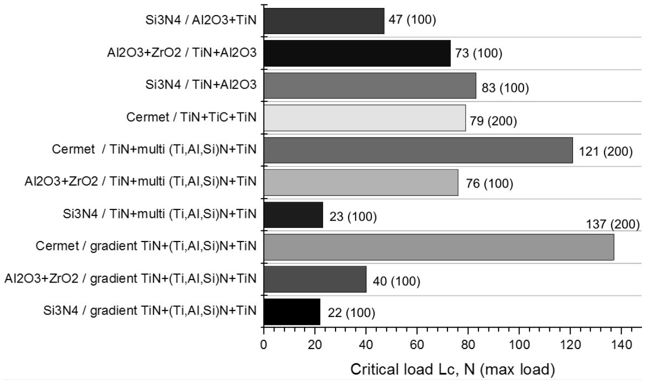
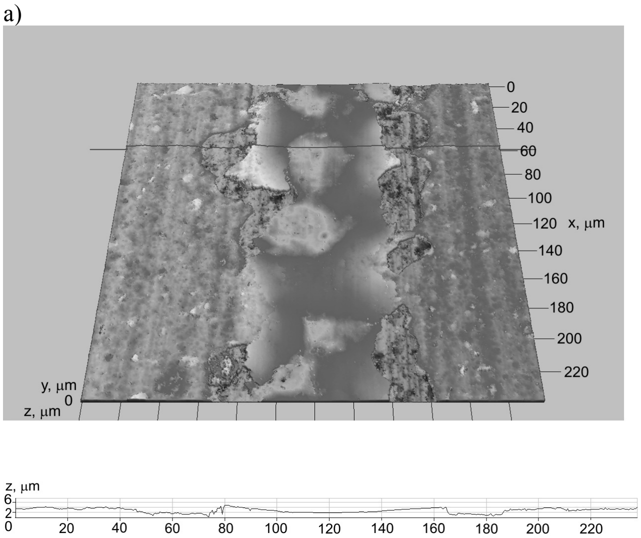


Fig. 2. Comparison of the critical load reached during scratch test

a)



b)

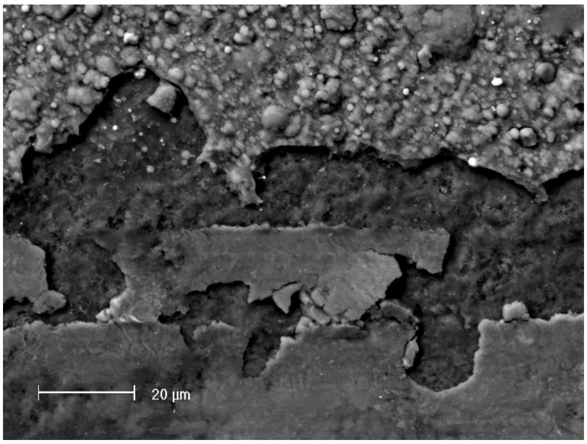


Fig. 3. Scratch test: Indenter trace of the TiN+TiAlSiN+TiN coating surface deposited onto the Si_3N_4 nitride ceramics substrate a) confocal microscope, b) SEM

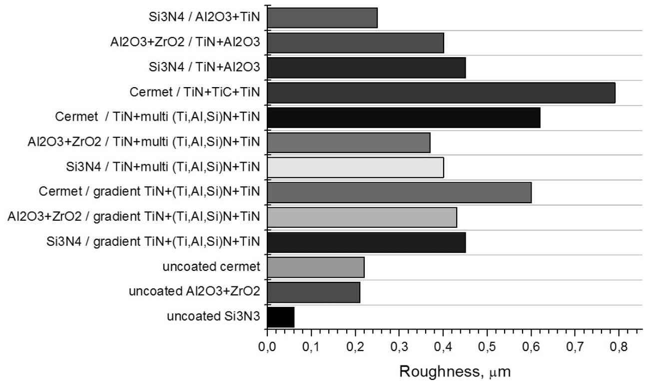


Fig. 4. Comparison of the roughness parameter R_a of the substrates and deposited coatings

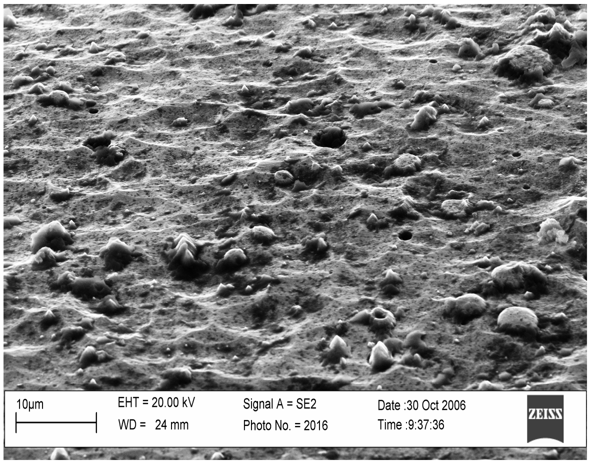


Fig. 5. Topography of the TiN+(Ti,Al,Si)N+TiN gradient coatings surface on the cermet substrate

Roughness of the uncoated substrates defined by R_a parameter is within 0.06-0.22 µm range and significantly increase after deposition of investigated coatings what should be attributed to the PVD process character and occurrence of the micro-droplets, coming from the sputtered disk (Figs. 4 5).

The existance of interlayers between Si_3N_4 substrate (for CVD coatings) and Al_2O_3+ZrO_2 and cermet substrates (for PVD coatings) and between layers in the coatings causes the increase of adherence and also the improvement of cutting ability of examined materials what was confirmed among others by reasearches made by the use of GDOES method. These researches confirm presence of titanium, aluminum, silicon and oxygen as well as nitrogen in the investigated coatings and of the substrate elements, among others titanium, nickel, cobalt, zirconium, silicon, carbon, nitrogen and oxygen (Fig. 6). The analyses show that in the interlayer the increase of concentration of elements being in the composition of the substrate from the coating surface appears at the concurrent decrease of elements creating coatings. It testifies about the existence of the interlayer between substrate material and the coating with earlier described diffusional migration of the chemical elements, having the influence on the improvement of adherence of deposited coatings to the substrate. However, the results cannot be interpreted equivocally because of heterogeneous material evaporation from the sample surface during the analysis. The existence of the interlayer can be explained also by the action of ions having high energy and causing the migration of elements in the interlayer, the increase of disorption of substrate surface and the appearance of defects in the substrate in the conditions of coating deposition, especially in the PVD process.

The researches on the SEM of the fracture of analised cermets and tool ceramics with coatings deposited by the PVD and CVD method do not show the delamination between the coating and substrate what also indicates the good adherence of achieved coatings. All PVD and CVD gradient-, two- and multilayer coatings deposited onto the nitride, oxide ceramics and cermets are characterised by a structure without pores and discontinuities and by tight adherence to themselves and of the entire multilayer coating to the substrate (Fig. 7).

Examinations of thin foils from coatings confirm that, according to the original assumptions, coatings containing the TiN type phases were deposited onto the cermets substrate. 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. Structure of the gradient TiN+(Ti,Al,Si)N+TiN coating, presented in Figure 8a, is fine grained and size of the TiN phase particles with the cubic lattice does not exceed 50 nm. Examination results of the thin foil from the transverse section of the TiN+Al_2O_3 coating indicate that the TiN coating has a columnar structure; whereas, the Al_2O_3 layer – the coarse-grained structure. There is an interface between the TiN and Al_2O_3 layers, where the fine grains of these phases are found (Fig. 8b).

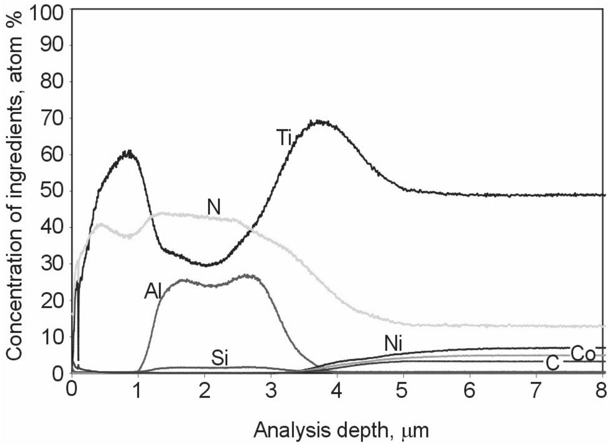


Fig. 6. Concentration changes of the TiN+(Ti,Al,Si)N+TiN gradient coating elements, substrate material from the cermet analysed in the GDOES spectrometer

As a result of carried out researches it was found out that the deposition of multilayer and gradient nanocrystalline coatings by the use of PVD and CVD method causes the increase of cutting properties of tools made of cermets and Al_2O_3+ZrO_2 comparing to adequately uncoated tools. The increase of cutting properties indicates also tools made of Si_3N_4 with two-layer TiN+Al_2O_3 coating, analogical to tools coated by the similar system of coatings available on the market.

Therefore, the real life tests (Fig. 9) confirm the quality of the III generation coatings of the TiN+ multi (Ti,Al,Si)N+TiN and TiN+gradient (Ti,Al,Si)N+TiN types obtained with the PVD technique in the cathode arc evaporation CAE process on the oxide and nitride ceramics and on tool cermets, as the material that significantly decreases the abrasive wear, thermal and adhesion wear, which immediately affects, among others, extension of the tool life, compared to the uncoated tools, and those with the multiple-layer coatings deposited using the CVD or PVD methods. The desired decrease of the particular wear types (abrasive, thermal, and adhesion ones) of the cutting tools, demonstrated by extension of the tool life, by deposition of the wear resistant coatings on their working surfaces, should be connected with a high micro-hardness of the coatings at the „room" temperature and at the elevated temperatures, with the low chemical affinity of cutting tool material to the machined material (mostly to iron and carbon) and with protecting the tool edge from oxidation and excessive overheating.

a)

b)

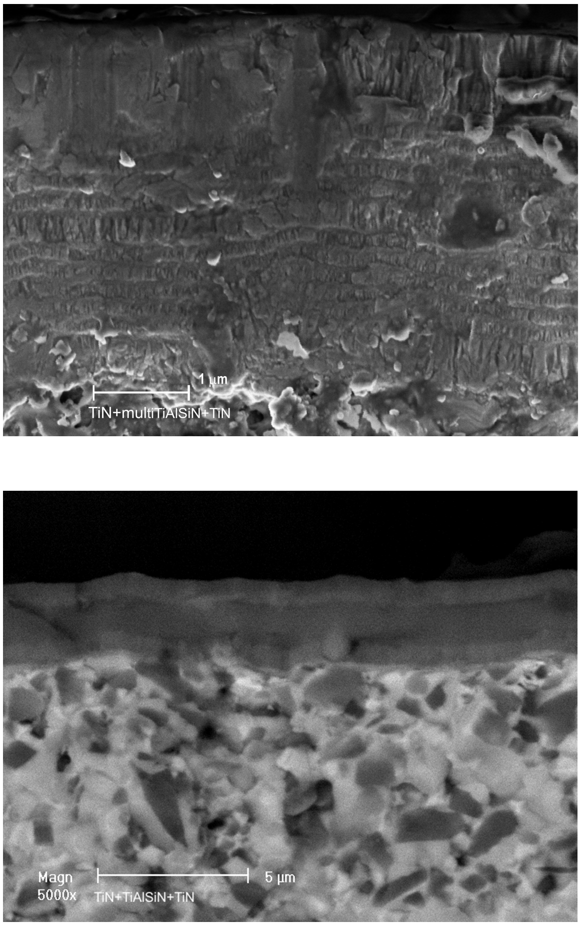


Fig. 7. Fracture surface of the: a) TiN+multi(Ti,Al,Si)N +TiN, b) TiN+(Ti,Al,Si)N+TiN gradient coating deposited onto the cermet substrate

a)

b)

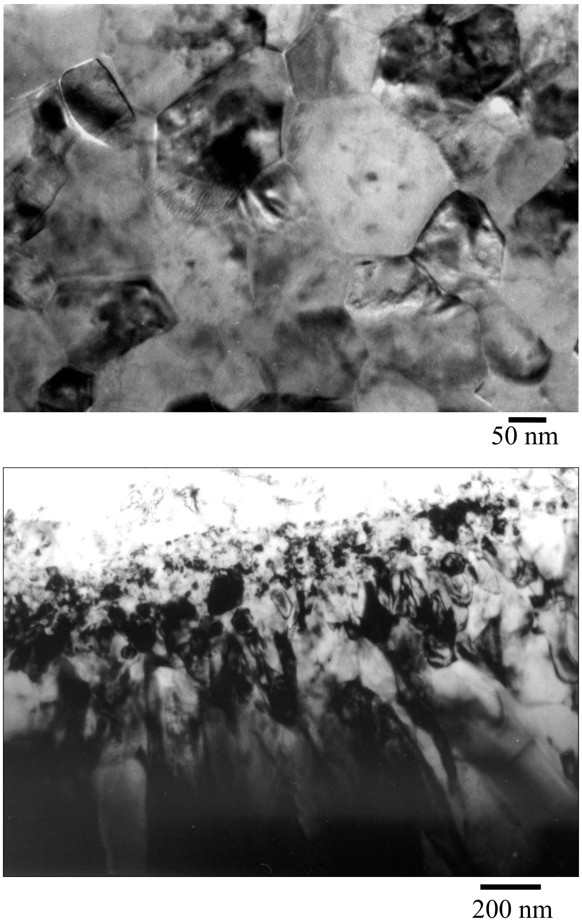


Fig. 8. Thin foil structure from the a) TiN+(Ti,Al,Si)N+ TiN gradient coating deposited on the cermet (TEM), b) TiN+Al_2O_3 coating (cross section; TEM)

4. Conclusions

As a result of carried out researches it was found out that the deposition of newly worked out multilayer and gradient TiN+(Ti,Al,Si)N+TiN nanocrystalline coatings by the use of PVD method and causes the increase of cutting properties of tools made of cermets for ca. 300% and of Al_2O_3+ZrO_2 for ca. 100% comparing to adequately uncoated tools. The increase of cutting properties for ca. 100% indicates also tools made of Si_3N_4 with two-layer TiN+Al_2O_3 coating, analogical to tools coated by the similar system of coatings available on the market.

In the case of commercial multilayer TiN+TiC+TiN coating on cermet substrate deposited with PVD process, the cutting ability are increased only for ca. 15%. As a result of little substrate roughness Ra=0.06 mm of Si_3N_4 ceramic inserts gradient and multilayer PVD coatings deposited on that substrate indicate little adherence L_c= ~20 N what explains lack of their influence on tool cutting properties. In the case of the rest of

a)

b)

c)

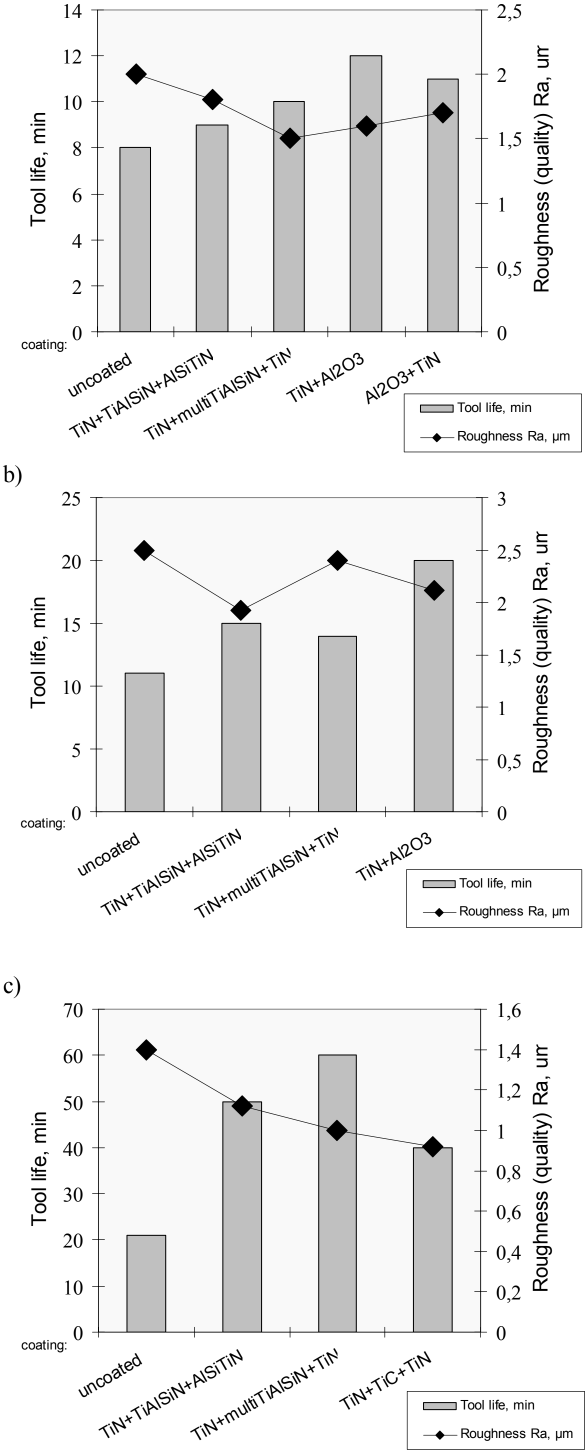


Fig. 9. Comparison of the values of the VB wear of the: a) Si_3N_4 ceramics, b) Al_2O_3+ZrO_2 ceramics, c) cermets uncoated and coated, depending on machining time and roughness of machined material

substrates having significantly bigger roughness Ra= ~0.2 mm good and very good adherence (L_c) respectively 80-140 N were achieved, what is connected with the creation of the interlayer between substrate and coatings and between layers of the coatings with fine grains structure identified in the research of thin foils on the TEM. However, the most profitable adherence shows the layers in which not only the good adhesion commonly described in the literature and also at least partially difussional character of the adherence between substrate and layer confirmed by the GDOES methods were stated.

Acknowledgements

Research was financed partially within the framework of the Polish State Committee for Scientific Research Project KBN PBZ-100/4/T08/2004 headed by Prof. L.A. Dobrzański.

References

[1] L.A. Dobrzański, K. Gołombek, J. Mikuła, D. Pakuła, Cutting ability improvement of coated tool materials, Journal of Achievements in Mechanical and Materials Engineering 17 (2006), 41-44.

[2] L.A. Dobrzański, D. Pakuła, Structure and properties of the wear resistant coatings obtained in the PVD and CVD processes on tool ceramics, Materials Science Forum 513 (2006) 119-133.

[3] L.A. Dobrzański, J. Mikuła, K. Gołombek, Structural characteristics of the modern sintered tool materials, Materials Science Forum 530-531 (2006) 499-504.

[4] L.A Dobrzański, J. Mikuła, The structure and functional properties of PVD and CVD coated Al_2O_3+ZrO_2 oxide tool ceramics, Journal of Materials Processing Technology 167 (2005) 438-446.

[5] D.V. Shtansky, I.V. Lyasotsky, N.B. D'yakonova, F.V. Kiryukhantsev-Korneev, S.A. Kulinich, E.A. Levashov, J.J. Moore, Comparative investigation of Ti–Si–N films magnetron sputtered using Ti Si qTi and Ti Si qTiN targets, Surface and Coatings Technology 182 (2004) 204–214.

[6] A. Jehn Hermann, Multicomponent and multiphase hard coatings for tribological applications, Surface and Coatings Technology 131 (2000) 433-440.

[7] S. Tjong, H. Chen, Nanocrystalline materials and coatings, Materials Science. and Engineering R45 (2004) 1–88.

[8] Y.Y. Tse, D. Babonneau, A. Michel, G. Abadias, Nanometer-scale multilayer coatings combining a soft metallic phase and a hard nitride phase: study of the interface structure and morphology, Surface and Coatings Technology 180-181 (2004) 470-477.

[9] M. Soković, J. Mikuła, L.A. Dobrzański, J. Kopač, L. Kosec, P. Panjan, A. Piech, Cutting properties of the Al_2O_3+SiC(w) based tool ceramic reinforced with the PVD and CVD wear resistant coatings, Journal of Materials Processing Technology 164-165 (2005) 924-929.

[10] L.A. Dobrzański, K. Gołombek, Structure and properties of the cutting tools made from cemented carbides and cermets with the TiN+mono-, gradient- or multi(Ti, Al, Si)N+TiN nanocrystalline coatings Journal of Materials Processing Technology 164-165 (2005) 805-815.

[11] P. Holubar, M. Jilek, M. Sima, Present and possible future applications of superhard nanocomposite coatings, Surface and Coatings Technology 133-134 (2000) 145-151.

[12] S. Ekinovic, Metal cutting, Faculty of Mech. Eng., Zenica, Bosnia & Hertzegovina, 2001 (in Bosnian).

[13] J. Kopac, Cutting tool wear during high-speed cutting. Stroj. vestn. Vol. 50 No. 4 (2004) 195-205.

[14] P. Panjan, I. Boncina, J. Bevk, M. Cekada, PVD hard coatings applied for wear protection of drawing dies. Surface and Coatings Technology 200 (2005) 133-136.

[15] M. Sokovic, L.A. Dobrzanski, J. Kopac, L. Kosec, Cutting Properties of PVD and CVD Coated Al_2O_3 + TiC tool ceramic, Proceedings of International Conference Thermec 2006 Vancouver, Canada.

[16] T. Wierzchoń, Structure and properties of multicomponent and composite layers produced by combined surface engineering methods, Surface and Coatings Technology 180 –181 (2004) 458–464.

[17] Dayan Ma, Shengli Ma, Kewei Xu, Influence of Si content on Nano-structured Ti–Si–N films coated by pulsed-d.c. plasma enhanced CVD, Surface and Coatings Technology 184 (2004) 182–187.

[18] S. PalDey, S.C. Deevi, Properties of single layer and gradient (Ti,Al)N coatings, Materials Science and Engineering A361 (2003) 1–8.