



of Achievements in Materials and Manufacturing Engineering VOLUME 41 ISSUES 1-2 July-August 2010

# Properties of Ti(B,N) coatings deposited onto cemented carbides and sialon tool ceramics

#### L.A. Dobrzański <sup>a,\*</sup>, M. Staszuk <sup>a</sup>, K. Gołombek <sup>a</sup>, M. Pancielejko <sup>b</sup>

 <sup>a</sup> 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
<sup>b</sup> Institute of Mechatronics, Nanotechnology and Vacuum Technique, Koszalin University of Technology, ul. Racławicka 15-17, 75-620 Koszalin, Poland
\* Corresponding author: E-mail address: leszek.dobrzanski@polsl.pl

Received 13.03.2010; published in revised form 01.07.2010

# **Properties**

# ABSTRACT

**Purpose:** The aim of this paper was to investigate mechanical properties both of sintered carbides WC-Co type and sialon tool ceramics with wear resistance ternary coatings Ti(B,N) type deposited by the cathodic arc evaporation process (CAE-PVD).

**Design/methodology/approach:** The microhardness tests of coatings were made using the ultra microhardness tester. The grain size of investigated coatings was determined by the Scherrer method. Tests of the coatings adhesion to a substrate material were made using the scratch test. There was investigated the roughness of both uncoated and coated surface multi-point inserts. Wear mechanism observations, after the scratch test, were carried out by the scanning electron microscope with EDS attachment.

**Findings:** This paper presents that studied PVD coatings deposited on sintered carbides and sialon tool ceramics have an effect on increasing hardness surface of tools. Moreover, the results achieved after the investigation shown that a coating obtaining on tool ceramics has bigger grains and a smaller adhesion to substrate rather than a coating on sintered carbides. Furthermore, the investigations were shown that both single and double-sided delamination was a principal defect mechanism during the scratch test.

**Practical implications:** The gradient Ti(B,N) coating carried out on multi point inserts (made on sintered carbides WC-Co type) can be used in the pro-ecological dry cutting processes without using cutting fluids. However, application of this coating to cover sialon ceramics demands still both elaborating and improvement adhesion to substrates in order to introduce these to industrial applications.

**Originality/value:** The paper presents some researches of gradient Ti(B,N) nanocrystaline coatings deposited by CAE-PVD method on sintered carbides and sialon tool ceramics.

Keywords: Mechanical properties; Wear resistance; Tool materials; Thin and hard coatings

#### Reference to this paper should be given in the following way:

L.A. Dobrzański, M. Staszuk, K. Gołombek, M. Pancielejko, Properties of Ti(B,N) coatings deposited onto cemented carbides and sialon tool ceramics, Journal of Achievements in Materials and Manufacturing Engineering 41/1-2 (2010) 66-73.

Properties

## **1. Introduction**

Cutting tools, which work in most cases in the conditions of load mining undergo a wear. One of the most important methods is a deposit both hard and against wear coatings, which increases a life operating of machine edges [1-16,18]. One of methods generally uses in the industrial usage is the cathodic arc evaporation CAE-PVD [1-3,6,7,16,18].

The paper presents the results of continuity researches of a ternary coating Ti(B,N) obtained on sintered carbides and sialon tool ceramics. The detail analysis of influence parameters on a obtained structure of this coating was presented in work [1]. The investigations presented in a previous work were shown that a obtained coating on sintered carbides and sialon tool ceramics, had a fine grained structure comply with a T zone and III zone according to a Thornton's model. The topography observation is shown occurrence of numerous drops of evaporated target, what is a disadvantage of obtained coatings. Both the analysis of phase composition by XRD method and the research of thin foil were shown mainly occurring isomorphic phase with a titanium nitride TiN [1].

#### 2. Experimental

The investigations were carried out on the multi-point inserts uncoated and coated by the Ti(B,N) gradient PVD coating. Inserts were made by both sintered carbides WC-Co and sialon tool ceramics. Coating was obtained by the cathodic arc evaporation method using a system Dreva Arc 400 (Fig. 1). Detailed conditions depositing process is given in the work [1].



Fig. 1. PVD system Dreva Arc 400

Thickness of coatings were measured with using the kalotest method. An average number of 5 craters were carried out in each sample in order to determine a coating thickness.

The roughness measurements  $R_a$  of both uncoated and coated samples surfaces of investigating coatings were carried out with using a profile measurement gauge Surftronic 3+ Taylor Hobson company. There was made an assumption of both a testing length  $L_c = 0.8$  mm and an accuracy measurement  $\pm 0.02$  µm. The microhardness of investigated materials was determined with using a Vickers method. The microhardness of substrates was measured with using a classic Vickers method. The applied load was equal 3N according to a PN-EN ISO 6507-1:2007 standard. The microhardness investigations were carried out with using a dynamic Vickers method in a way of load – unload. Applied load was not exceed 0.5 N, what rejects an influence substrate on a measurement result of micro hardness coating.

The substrate adhesion to substrate was determined on the basis on investigation of the Scratch Test using a Revetest device of CSEM company. There were applied the following investigation parameters such as: a load range 0 - 100 N, a rate of increment load 100 N/min, a shift indenter rate 10 mm/min, a sensitivity detector acoustic emission equals 1.

The observation of formed scratch as a result of the Scratch Test was carried out with using the scanning electron microscope DSM 940 of Opton company.

The chemical concentration changes of the coating components, both perpendicular to their surface and in the transition zone between the coating and the substrate material were evaluated in virtue of tests carried out in the GDOS-750 QDP glow discharge optical spectrometer from Leco Instruments. The following operation conditions of the spectrometer Grimm lamp were fixed during the tests: lamp inner diameter -4 mm; lamp supply voltage -700 V; lamp current -20 mA; working pressure -100 Pa.

### <u>3. Results</u>

The results of thickness investigations obtained coatings are presented in Table 1. It was found that a coating obtained on substrate from sintered carbides shows bigger thickness rather than a coating obtained on substrate from sialon ceramics. The reason of getting thicker coating on sintered carbides is a possibility of connect a accelerating voltage to substrate of this type. The connecting voltage bias causes bigger rate of increasing coating on sintered substrates rather than on does not polarized ceramic substrate.

As result of microhardness investigations of sintered carbides and sialon ceramics, both uncoated and coated investigated coatings it was found that microhardness growth of surface layer multi-point inserts after deposited investigating coatings (Table 2, Fig. 2). It was found too that microhardness of coatings obtained on sintered carbides is higher rather than on sialon ceramics. This is indubitable bound up with a structure obtained coatings. As it was shown in work [1] the structure of obtained coating Ti(B,N) on sintered carbides substrate is ultra-fine-grained and comply with a (IV) according to the Thornton's model. However, the coat structure obtained on sialon shows a structure comply with a III zone according to the Thornton's model. The differences into

	Ta	ble	1.		
--	----	-----	----	--	--

Thickness of investigated coatings

Substrate	Coating	Thickness, µm
Sintered carbides	Ti(D N)	1.8
Sialon ceramics	11( <b>D</b> ,1 <b>N</b> )	1.3

Grain size of investigated coatings and micronardness of investigated samples							
Substrate	Coating	Grain size,	Microhardness, HV			Standard	Confidence
			min. value	max. value	mean value	deviation	$\alpha=0.05$
Sintered	Uncoated	-	1789	1865	1826	26.87	±21.84
carbides	Ti(B,N)	21	2633	3049	2951	158.52	$\pm 128.83$
Sialon	Uncoated	-	1990	2080	2035	31.81	±25.86
ceramics	Ti(B,N)	57	2284	3067	2676	356.85	±290.01

Table 2. Grain size of investigated coatings and microhardness of investigated sample

Table 3.

· · · · · · · · · · · · · · · · · · ·	Critical load and	roughness	of investigated	samples
---------------------------------------	-------------------	-----------	-----------------	---------

Substrate	Coating	Coating Critical load	Roughness, R <sub>a</sub> , μm			Standard	Confidence
Substrate	Coating		min. value	max. value	mean value	deviation	$\alpha = 0.05$
Sintered	Uncoated	-	0,06	0,06	0,06	0	$\pm 0$
carbides	Ti(B,N)	34	0.20	0.46	0.29	0.12	±0.13
Sialon	Uncoated	-	0,06	0,06	0,06	0	$\pm 0$
ceramics	Ti(B,N)	13	0.18	0.34	0.25	0.06	±0.07

a structure of both coatings it is necessary to connect with a possibility of polarization substrate, what was written in paper [1]. These results confirm also a measurement of size grain realized by the Scherer's method. This test confirms that a Ti(B,N) coating obtained on sintered carbides presented smaller size grains (21 nm) in comparison to a coating obtained on sialon ceramics (57 nm) (Table 2, Fig. 2).

It was found that an increase of surface roughness, after deposited coatings (Table 3) on the basis of surface roughness investigations  $R_a$  multi-point inserts on sintered carbides and sialon ceramics, both uncoated and uncoated inserts. The increase of surface roughness is connect with a occurrence solidified drops of metal (Fig. 4), what is a characteristic feature for coatings obtained by the cathodic arc evaporation method.

The value of critical load which is a measure of adhesion coatings to surface from sialon ceramics and sintered carbides was determined by the Scratch Test (Table 3, Fig. 3). In a case of obtained coatings Ti(B,N), it was not possible to determine a critical load on basis of acoustic emission AE figure



Fig. 2. The comparison between microhardness inserts on sintered carbides and sialon ceramics, both uncoated and deposited Ti(B,N) coating and a grain size investigated coating

(Figs. 6, 7). In the consequence of this the critical load was determined on the basis of scratch observation, which was formed during a test. Observation was realized onto light microscope coupling with a measurement device. As a result of investigation it was found that a Ti(B,N) coating presents better adhesion to substrate on sintered carbides rather than on sialon ceramics. Both a adhesion and also a small diffusion displace of chemical elements inside a connection zone (which is a result of ion implantation about huge energy falling on a negative polarized substrate) have an effect on coating adhesion to substrate on sintered carbides. The investigation results obtained with using the glow discharge optical spectrometer confirm this (Fig. 5). High of energy ions falling on polarized substrate causes numerous phenomena's such as: local increase of temperature, acceleration а of chemisorptions, intensification of surface diffusion processes and in the surface depth, there can also occur a ion penetration into not a big depth (about a few nm) and partial atomization of atoms a depositing coating [17,18].



Fig. 3. The comparison between critical loads  $L_c$  and thickness of coatings deposited on sintered carbides and sialon ceramics



Fig. 4. Surface topography of the  $\mathrm{Ti}(\mathrm{B},\mathrm{N})$  coating deposited on cemented carbide





Fig. 5. Changes of constituent concentration of the Ti(B,N) coating and a sintered carbide substrate material



Fig. 6. a) Acoustic emission (AE) and friction force Ft as a function of the normal load Fn for the Ti(B,N) coating on sintered carbides, b) the Scratch failure pictures of the Ti(B,N) coating on sintered carbides at  $L_c$  (opt) = 34 N, mag. 200x



Fig. 7. a) Acoustic emission (AE) and friction force Ft as a function of the load Fn for the Ti(B,N) coating on sialon ceramics, b) the Scratch failure pictures of the Ti(B,N) coating on sialon ceramics at  $L_c$  (opt) = 13 N, mag. 200x



Fig. 8. a, b, c) Characteristic failure of the Ti(B,N) coating deposited on sintered carbides developed during the adhesion scratch test, d) X-ray energy dispersive plot the area X1 as in figure c, e) X-ray energy dispersive plot the area X2 as in a figure c, f) X-ray energy dispersive plot the area X3 as in a figure c

# **Properties**



Fig. 9. a, b) Characteristic failure of the Ti(B,N) coating deposited on sialon ceramics developed during the adhesion scratch test, c) X-ray energy dispersive plot the area X1 as in a figure b, d) X-ray energy dispersive plot the area X2 as in a figure b. Maps of superficial distribution chemical elements from areas as in a figure a: e) Ti - area with a coating, f) Si - area without a coating

Adhesion investigations of coatings to substrate by the Scratch Test simulates some conditions occurred during a machining. This research takes into account an influence on normal force and a friction force. The observation results of damages formed as a result of adhesion investigations by the Scratch Test are presented in Figures 8 and 9. The single and double-sided delamination (occurs after exceed a critical load) is a dominant mechanism of damaged coating. Furthermore, it was found a dent in a central side of scratch in a case of coating obtained on sintered carbides. The cracks in a shape of arc are caused by coating tension during an investigation. It was not found any a plastic strain of coating in a case of obtained coating on sialon ceramics, but only some brittle spallings of coating. This is undoubtedly connected with a higher substrate hardness and lower thickness and lower adhesion of coatings to a ceramic substrate. Observed mechanism of damages in each cases was confirmed by realized EDS analysis (Figs. 8 d, e, f, 9 c, d, e, f).

### 4. Summary

The investigation results of obtained coating properties Ti(B,N) by the cathodic arc evaporation method on substrates on both sintered carbides and tool sialon ceramics. Micro hardness coated multi-point inserts distinct increased and was in the range from 2676 to 2951 HV. In a case of coating on sintered carbides the hardness increase of surface layer in compare with a substrate without coat is equal 60%. It was found a surface roughness increase after deposited a coating what was connected with a occurrence of evaporated solidify metal drops on the samples surface. The adhesion investigations of coatings to substrate on sintered carbides rather than on sialon ceramics. This has undoubtedly a connection with a possibility polarization of carbides substrate and consequently lower thickness of coatings.

Numerous investigations [3,5,12,19] show that loss of coating adhesion to a substrate is not necessity connect with a coating removal. The investigations presented in this paper show that investigated coating was expired both single and double sided delimitation, but inside a scratch was indented in a substrate a coating material. In a case of coating on sintered carbides it shows some tracks of plastic tension, but in a case of coating on sialon ceramics there are shown on it some brittle spalling.

#### Acknowledgements

The research was financed partially within the framework of the research project N N507 493438 of the Polish State Committee for Scientific Research, headed by Dr Daniel Pakuła.

The paper has been realised in relation to the project POIG.01.01.01-00-023/08 entitled "Foresight of surface properties formation leading technologies of engineering materials and biomaterials" FORSURF, co-founded by the European Union from financial resources of European Regional Development Found and headed by Prof. L.A. Dobrzański.



#### <u>References</u>

- L.A. Dobrzański, M. Staszuk, J. Konieczny, W. Kwaśny, M. Pawlyta, Structure of TiBN coatings deposited onto cemented carbides and sialon tool ceramics, Archives of Materials Science and Engineering 38/1 (2009) 48-54.
- [2] L.A. Dobrzański, M. Staszuk, M. Pawlyta, W. Kwaśny, M. Pancielejko, Characteristics of Ti(C,N) and (Ti,Zr) N gradient PVD coatings deposited onto sintered tool materials, Journal of Achievements in Materials and Manufacturing Engineering 31/2 (2008) 629-634.
- [3] L.A. Dobrzanski, K. Gołombek, J. Mikuła, D. Pakuła, Multilayer and gradient PVD coatings on the sintered tool materials, Journal of Achievements in Materials and Manufacturing Engineering 31/2 (2008) 170-190.
- [4] W. Kwaśny, Predicting properties of PVD and CVD coatings based on fractal quantities describing their surface, Journal of Achievements in Materials and Manufacturing Engineering 37/2 (2009) 125-192.
- [5] K. Gołombek, J. Mikuła, D. Pakuła, L.W. Żukowska, L.A. Dobrzański, Sintered tool materials with multicomponent PVD gradient coatings, Journal of Achievements in Materials and Manufacturing Engineering 31/1 (2008) 15-22.
- [6] L.A. Dobrzański, K. Gołombek, Gradient coatings deposited by Cathodic Arc Evaporation: characteristic of structure and properties, Journal of Achievements in Materials and Manufacturing Engineering 14 (2006) 48-53.
- [7] 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.
- [8] W.Y.H. Liew, Ultra-precision machining of stainless steel using coated carbide tool, Archives of Materials Science and Engineering 31/2 (2008) 117-120.
- [9] H. Holzschuh, Deposition Ti-B-N (single and multilayer) and Zr-B-N coatings by chemical vapor deposition techniques on cutting tools, Thin Solid Films 469-470 (2004) 92-98.
- [10] M.J. Jackson, G.M. Robinson, J.S. Morrell, Machining M42 tool steel using nanostructured coated cutting tools, Journal of Achievements in Materials and Manufacturing Engineering 23/1 (2007) 83-86.
- [11] J. Kopač, Influence of cutting material and coating on tool quality and tool life, Journal of Materials Processing Technology 78 (1998) 95-103.
- [12] D. Pakuła, L.A. Dobrzański, K. Gołombek, M. Pancielejko, A. Křiž, Structure and properties of the Si<sub>3</sub>N<sub>4</sub> nitride ceramics with hard wear resistant coatings, Journal of Materials Processing Technology 157-158 (2004) 388-393.
- [13] H.G. Prengel, P.C. Jindal, K.H. Wendt, A.T. Santhanam, P.L. Hegde, R.M. Penich, A new class of high performance PVD coatings for carbide cutting tools, Surface and Coatings Technology 139 (2001) 25-34.
- [14] M. Soković, J. Mikuła, L.A. Dobrzański, J. Kopač, L. Koseč, P. Panjan, J. Madejski, A. Piech, Cutting properties of the

# Properties

 $Al_2O_3 + SiC_{(w)}$  based tool ceramic reinforced with the PVD and CVD wear resistant coatings, Journal of Materials Processing Technology 164-165 (2005) 924-929.

- [15] M.J. Jackson, G.M. Robinson, J.S. Morrell, Machining strain hardening metals using nanostructured thin film end mills, Journal of Achievements in Materials and Manufacturing Engineering 21/1 (2007) 79-82.
- [16] Z. Werner, J. Stanisławski, J. Piekoszewski, E.A. Levashov, W. Szymczyk, New types of multi-component hard coatings deposited by ARC PVD on steel pre-treated by pulsed plasma beams, Vacuum 70 (2003) 263-267.
- [17] A. Michalski, Physical chemistry the basics of obtaining coatings from gaseous phase, Publishing House of Warsaw University of Technology, Warsaw, 2000 (in Polish).
- [18] K. Gołombek, Structure and properties sintered carbides and sialon ceramics and tool ceramics coated with coatings resistant on abrasion resistance in the PVD process, Ph.D. thesis (has not published), The Central Library of Silesian University of Technology, Gliwice, 2001 (in Polish).
- [19] M. Kupczyk, Analysis of theoretical patterns of Scratch Test adhesion test, Archives of Mechanical Technology and Automation 20/2 (2000) 167-179 (in Polish).