



# Investigations of the structure and properties of PVD coatings deposited onto sintered tool materials

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## ABSTRACT

**Purpose:** The paper presents investigation results of the structure and properties of the coatings deposited by cathodic arc evaporation - physical vapour deposition (CAE-PVD) techniques on the sialon tool ceramics. The Ti(B,N), Ti(C,N), (Ti,Zr)N, (Ti,Al)N and multilayer (Al,Cr)N+(Ti,Al)N, (Ti,Al)N+(Al,Cr)N coatings were investigated.

**Design/methodology/approach:** The structural investigation includes the metallographic analysis on the scanning electron microscope. Examinations of the chemical compositions of the deposited coatings were carried out using the X-ray energy dispersive spectrograph EDS. The investigation includes also analysis of the mechanical and functional properties of the material: microhardness tests of the deposited coatings, surface roughness tests, evaluation of the adhesion of the deposited coatings and tribological test made with the „pin-on-disk”.

**Findings:** Deposition of the multicomponent coatings with the PVD method, on tools made from sialon's ceramics, results in the increase of mechanical properties in comparison with uncoated tool materials, deciding thus the improvement of their working properties.

**Practical implications:** The multicomponent coating carried out on multi point inserts (made on sintered sialon's ceramics) 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 multicomponent coatings deposited by PVD method on sialon tool ceramics.

**Keywords:** Thin and thick coatings, Tool materials; PVD; Multicomponent coatings

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## MATERIALS MANUFACTURING AND PROCESSING

## 1. Introduction

Out of numerous types of tool materials, tool ceramics raise a growing interest in academic and industrial environments. This includes  $\beta$ -sialon ceramics developed in late 20<sup>th</sup> century. The mechanical properties of the alloy ceramics are from isomorphous  $\beta$ - $\text{Si}_3\text{N}_4$ , while the chemical properties correspond to  $\text{Al}_2\text{O}_3$  aluminium oxide. Hard PVD and CVD coatings applied on machining blades are effective means increasing the durability of tools from high speed steels and sintered carbides, used since 1960's. The opinion, common until recently, that ceramic tool coating is pointless, due to their generic hardness, has also been adjusted lately. Ceramic tools with abrasion-resistant coatings have been launched on the market recently. Wear-resistant coatings, based on nitrides, carbides, oxides and borides, of transition metals, mainly, enable the use of higher machining parameters of the tools coated with them and they also enable processing without the use of coolants - lubricants. In order to increase the durability, the ceramic tool materials are subjected to additional surface processing performed in CVD (Chemical Vapour Deposition) - and PVD (Physical Vapour Deposition) - applied more and more frequently at present. The tools with coatings based on carbides, borides, nitrides and oxides may be used for higher working parameters. The operating properties of the machining tools with hard anti-wear coatings are usually characterised with several times higher durability of the machining blade as compared to the uncoated tools [1-33].

The goal of this work is investigation of structure and properties of the PVD coatings deposited onto the sialon tool ceramics substrate.

## 2. Material and methods

The tests were carried out on multi-blade plates made from sialon tool ceramics, uncoated and coated in PVD process with multi-layer and gradient abrasion-resistant coatings (Table 1). The plates were coated in the CAE-PVD [cathodic arc evaporation - physical vapour deposition] process with coatings type  $\text{Ti}(\text{B},\text{N})$ ,  $(\text{Ti},\text{Zr})\text{N}$ ,  $(\text{Ti},\text{Al})\text{N}$ ,  $(\text{Al},\text{Cr})\text{N}$ ,  $(\text{Al},\text{Cr})\text{N}+(\text{Ti},\text{Al})\text{N}$  (Table 1).

The topography of surface and structure of the coatings produced on the transverse fractures was viewed in the scanning electron microscope Supra 35 from Zeiss. Secondary Electrons SE detection was used for obtaining the images of the samples tested, 5-20 kV acceleration voltage with maximum enlargement of 60000 times. The fragile fractures were prepared for viewing by means of carving notches on the plates with the coatings tested and, prior to breaking, they were subjected to cooling in liquid nitrogen. The qualitative and quantitative analyses of the chemical composition in the microspaces of the coatings tested were carried out by means of Energy Dispersive Spectroscopy (EDS) using EDS LINK ISIS from Oxford representing an accessory of Zeiss Supra 35 Scanning Electron Microscope. The tests were carried out at accelerating voltage of 20 kV.

The topography of the surface coatings obtained on sialon tool ceramics was also tested on Park Systems XE-100 atomic force microscope in touchless operating mode. The area of  $20 \times 20 \mu\text{m}^2$  of  $256 \times 256$  definition was tested.

The hardness of the materials tested was determined with the use of the Vickers method. The hardness of the bases from sialon tool ceramics were tested with the classic Vickers method, applying load equal to 3 N according to PN-EN ISO 6507-1:2007.

The adherence of the coatings to the base was assessed upon scratch test on Revetest device from CSEM. The method consists in moving Rockwell C diamond indenter through the surface of the sample tested at constant speed with applied force growing linearly. The following testing parameters were applied:

- the load range applied: 0-100 N,
- the load growth rate: 100 N/min,
- the indenter motion velocity: 10 mm/min,
- the acoustic emission detector's sensitivity: 1.

The abrasion-wear resistance and friction coefficient tests on the coatings with the „pin-on-disc” method were carried out on the CSEM device that is directly connected to the computer enabling the definition of the load extent, rotation velocity, radius on the sample, maximum friction coefficient, test duration. A WC [wolfram carbide] ball was used as a counter-sample. The tests were made in ambient room temperature, applying the following test results:

- pressure force  $F_N = 10 \text{ N}$ ;
- motion velocity  $v = 0.1 \text{ m/s}$ ;
- radius  $r = 5 \text{ mm}$ .

For all the samples tested the same number of cycles, i.e. 10000 was established.

Technological machining tests were carried out in order to sort the machining plates tested by their usable properties. The machinability of the uncoated and PVD sialon ceramic plates was tested based on a trial continuous turning without using the processing cooling-lubricating liquids on the TUR 630M lathe. The EN-GJL-250 grey cast iron of ca. 215 HB hardness.

The thin film structure observations and diffraction tests were carried out in JEM 3010 UHL electron transmission microscope from JEOL at 300 kV acceleration voltage and maximum enlargement 250 000 times. The diffractograms from the electron transmission microscope were solved with the use of „Eldy” software.

The continuous turning trials were carried out on multi-blade plates, type SNGN 120412 mounted on a universal lathe chuck.

The technological turning trials were carried out assuming the following parameters:

- feed  $f = 0.2 \text{ mm/rotation}$ ,
- turning depth  $a_p = 1 \text{ mm}$ ,
- machining velocity  $v_c = 180 \text{ m/min}$ .

The durability of the plates tested was determined in virtue of wear band width on the flank face. The mean wear band width VB and maximum wear band  $\text{VB}_{\text{max}}$  was carried out with the use of light microscope from Carl Zeiss Jena. The machining trials were stopped whenever the assumed wear criterion for finishing processing  $\text{VB} = 0.2 \text{ mm}$  was exceeded.

Table 1.  
Types of coatings obtained and used for tests and their basic properties

| Coatings          | Thickness of coating, $\mu\text{m}$ | Hardness HV0.5 | Roughness factor $R_a$ , $\mu\text{m}$ | Critical loading $L_c$ , N | Tool life $T$ , min |
|-------------------|-------------------------------------|----------------|--|----------------------------|---------------------|
| -                 | -                                   | 1838           | 0.06                                   | -                          | 11                  |
| Ti(B,N)           | 1.3                                 | 2676           | 0.25                                   | 13                         | 5                   |
| (Ti,Zr)N          | 2.3                                 | 2916           | 0.40                                   | 21                         | 5.5                 |
| (Al,Cr)N          | 4.8                                 | 2230           | 0.31                                   | 53                         | 50                  |
| (Ti,Al)N          | 5.0                                 | 2961           | 0.28                                   | 21                         | 9                   |
| (Al,Cr)N+(Ti,Al)N | 3.9                                 | 2558           | 0.44                                   | 69                         | 27                  |

### 3. Discussion of test results

The fractographic tests made in the scanning electron microscope indicate that the PVD coatings obtained are evenly laid and tightly adhere to the base (Fig. 1). Furthermore, the individual layers in the multilayer coating (Al,Cr)N+(Ti,Al)N present a compact structure, without delaminations or defects, and they tightly adhere to each other [27].

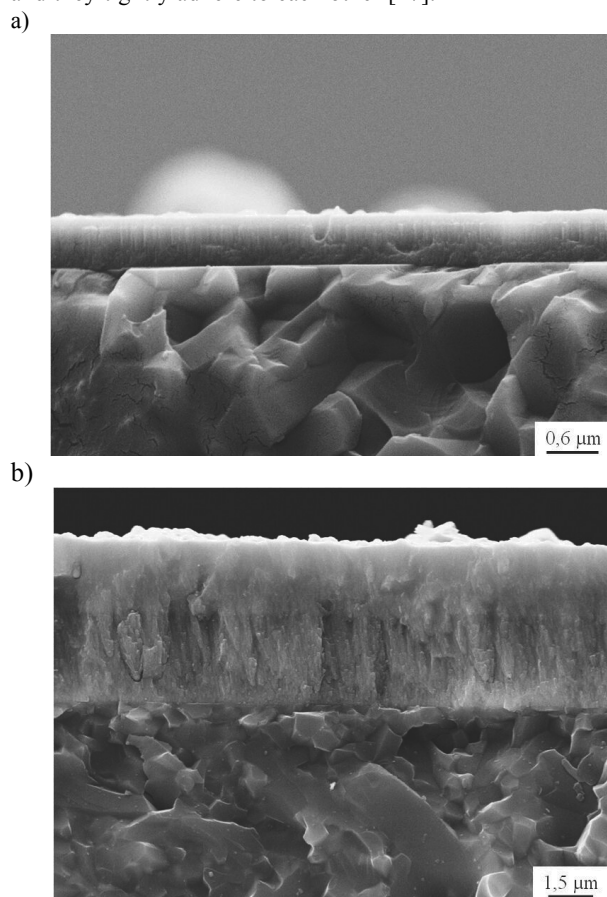


Fig. 1. Surface of coatings fracture: a) Ti(B,N) and b) (Al,Cr)N laid on sialon ceramics

Upon watching the fractures of PVD coatings, it was also found that the (Ti,Al)N, (Al,Cr)N+(Ti,Al)N and (Al,Cr)N

coatings present a structure classified within T zone, according to Thornton's model (Fig. 1b).

As a result of tests on thin films in the transmission electron microscope the fine granularity of the coatings tested was confirmed, while the electron diffractions confirmed the occurrence of the phases in the coatings tested - TiN and AlN for (Al,Ti)N layers and (Al,Cr)N (Fig. 2).

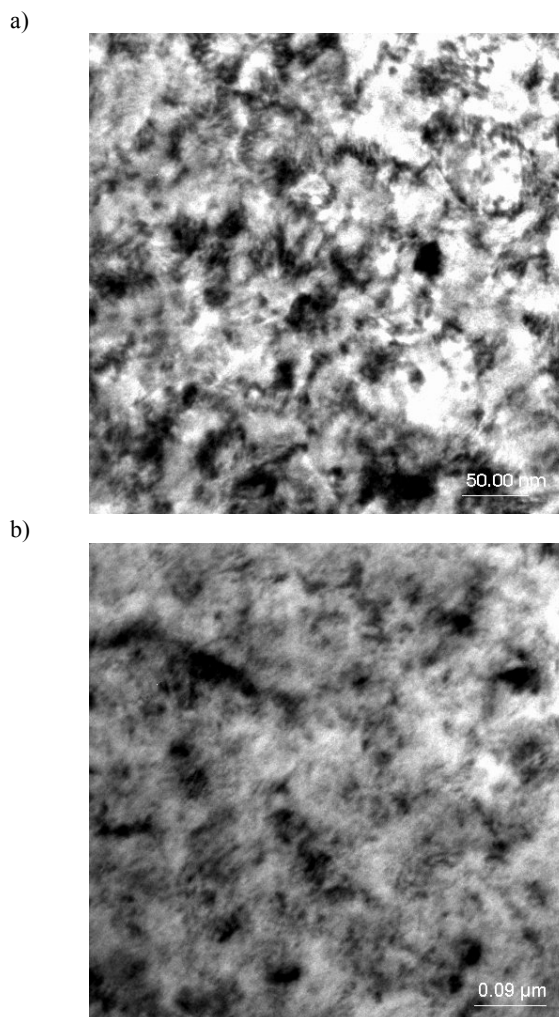


Fig. 2. Thin film structure of coating a) Ti(B,N), b) (Al,Ti)N



The thickness of the PVD coatings obtained on sialon tool ceramics is within the range 1.3 and 5.0  $\mu\text{m}$  (Table 1). Based on the roughness tests  $R_a$  of the surface of multi-blade plates from sialon tool ceramics uncoated and coated with the coatings tested, a growing roughness of the surface after laying the coating was observed, which is related to the inhomogeneity of the coatings' surface with numerous set metal drops and cavities. Roughness  $R_a$  of the multiblade plates' surface, PVD coated ranges from 0.15 to 0.50  $\mu\text{m}$ , while roughness coefficient of the uncoated base is  $R_a = 0.06 \mu\text{m}$  (Table 1).

Microhardness (Table 1) of the sialon tool ceramics tested is 1838 HV. Laying the PVD coatings causes significant growth of microhardness of the multi-blade plates' surface. The (Ti,Al)N coating shows the highest hardness, its micro-hardness is 2961 HV, which is ca. 60% of the surface layer hardness growth.

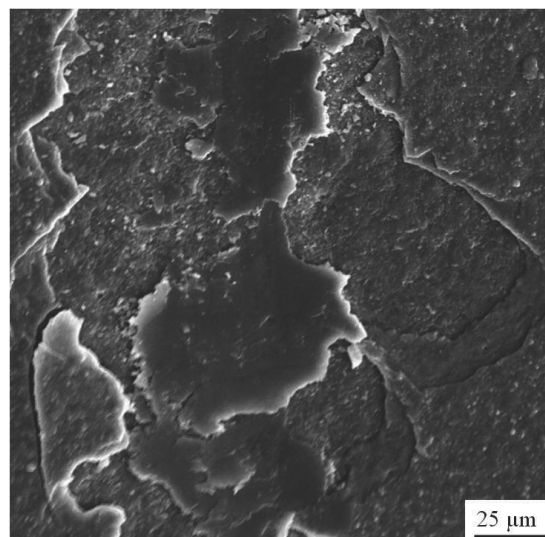
The critical load value  $L_c$  being the measure of PVD coatings adherence to the sialon tool ceramic base was determined upon a scratch-test. The critical load was settled as corresponding to acoustic emission growth signalling the beginning of the coating chipping and the verification was made basing on metallographic observations in the light microscope coupled with the measuring device. In case of PVD coatings obtained on sialon ceramics, the highest critical load value  $L_c = 69 \text{ N}$  is presented by two-layer coating (Al,Cr)N+(Ti,Al)N, while the lowest  $L_c = 13 \text{ N}$  by Ti(B,N) coating. Furthermore, it should be emphasised that (Al,Cr)N coating (Table 1) presents equally high critical load value  $L_c = 53 \text{ N}$  towards the sialon base. As a result of the tests it was found that there are 2 types of dominating damage mechanisms accompanied to a lesser extent by other symptoms. The first principal mechanism of coating damage observed upon exceeding the critical load is unilateral and bilateral delamination, which mainly concerns coatings Ti(B,N), (Ti,Al)N, (Al,Cr)N, (Al,Cr)N+(Ti,Al)N (Fig. 3a). Another damage mechanism occurring in case of coating type (Ti,Zr)N is abrasion accompanied by cohesive cracking of coatings and fine chipping and scaling (Fig. 3b).

The wear abrasion resistance „pin-on-disc” tests suggest that PVD coatings have good tribological properties. In almost all the coatings tested the coating is damaged down to the sialon ceramic base zone (Figs. 4, 6). The coating damages are accompanied by extensive adherence defects. The most frequent mechanisms of coating wear are chipping, scaling and delamination. The coatings with (Al,Cr)N layers have very good tribological properties, such layers are not damaged or, if any damages occur - they are slight. For all the PVD coatings tested, the damaged coating and counter-sample material adhere, which directly affects the variable friction coefficient values. The counter-sample material adheres to the (Al,Cr)N coatings most, which was confirmed on the EDS diagrams of the micro-area of the coating tested (Fig. 4). In case of this group of coatings the friction coefficient value varies depending on adherence of the damaged coating and counter-sample material applied. For gradient coatings Ti(B,N), (Ti,Zr)N, (Ti,Al)N and (Al,Cr)N and two-layer (Al,Cr)N+(Ti,Al)N the friction coefficient ranges within 0.4 and 0.7 (Figs. 5, 7).

The usable characteristics of the tested coatings obtained on the sialon ceramic blades was made basing on technological trials of continuous grey cast iron turning without the use of processing cooling-lubricating liquids. As a result of this test it was found

that the longest service time  $T = 50 \text{ min.}$  at machining velocity  $v_c = 170 \text{ m/min}$  was obtained for a (Al,Cr)N coated blade, while the lowest,  $T = 5 \text{ min}$  is for Ti(B,N) coatings. The life of uncoated sialon ceramic blade at the same machining velocity was estimated as  $T = 11 \text{ min}$  of continuous turning, which enables a statement that (Al,Cr)N, (Al,Cr)N+(Ti,Al)N coatings contribute to the increased durability of sialon blade (Table 1).

a)



b)

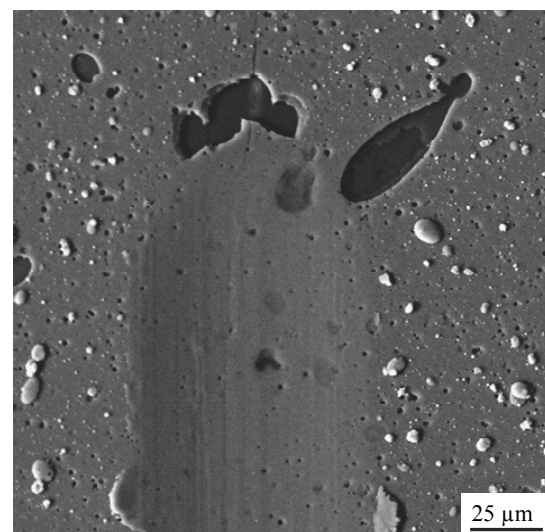


Fig. 3. View of damages produced as a result of a scratch-test of coating: a) (Al,Cr)N+(Ti,Al)N and b) (Ti,Zr)N made on sialon tool ceramics.

As a result of metallographic viewing in a scanning electron microscope of the multi-blade plates, it was found that the tools subjected to trial machining present wear according to the abrasive and adhesion mechanisms. The scaling of Ti(B,N), (Ti,Zr)N was observed, furthermore a limited buildup of the machined material occurred (Fig. 8).

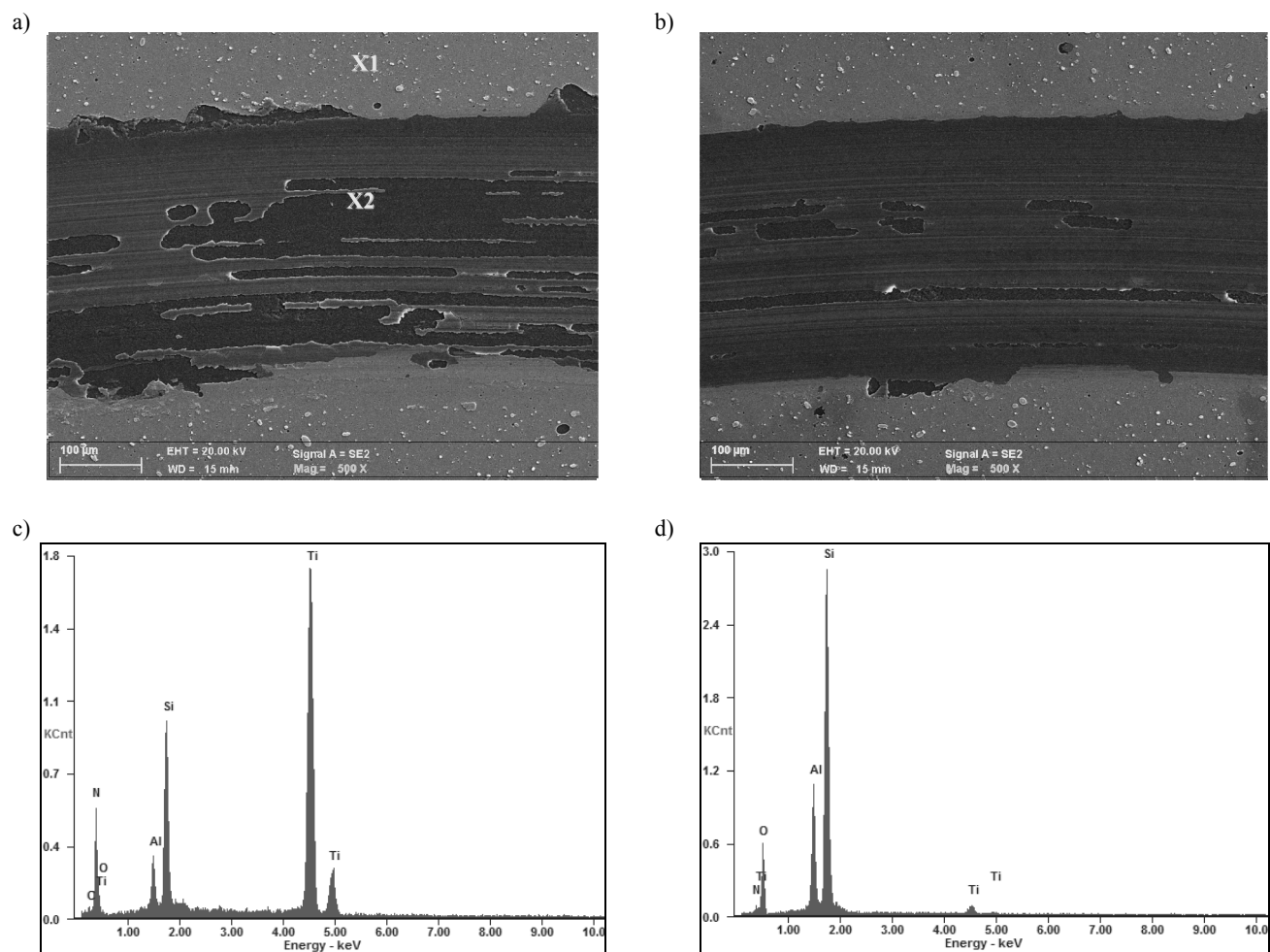


Fig. 4. a),b) Trace of tribological damage on Ti(B,N) coating surface on sialon ceramic base and energy diagrams of EDS from micro-area: c) X1, d) X2

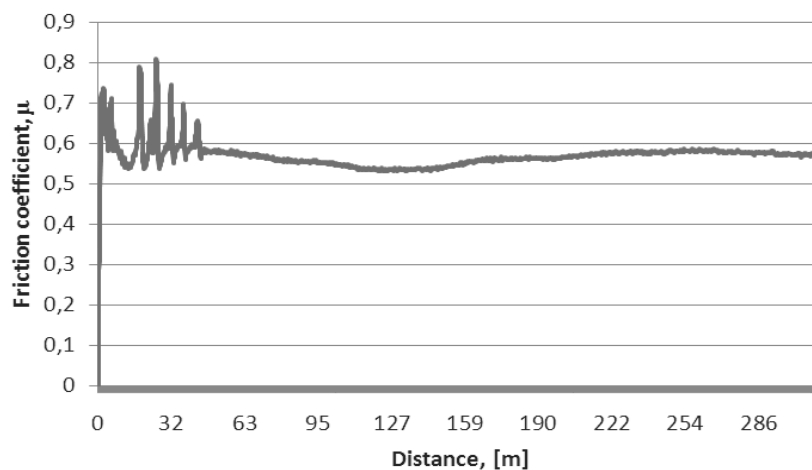


Fig. 5. Friction coefficient diagram depending on friction path during the „pin-on-disc” test for Ti(B,N) coating laid on sialon ceramic base

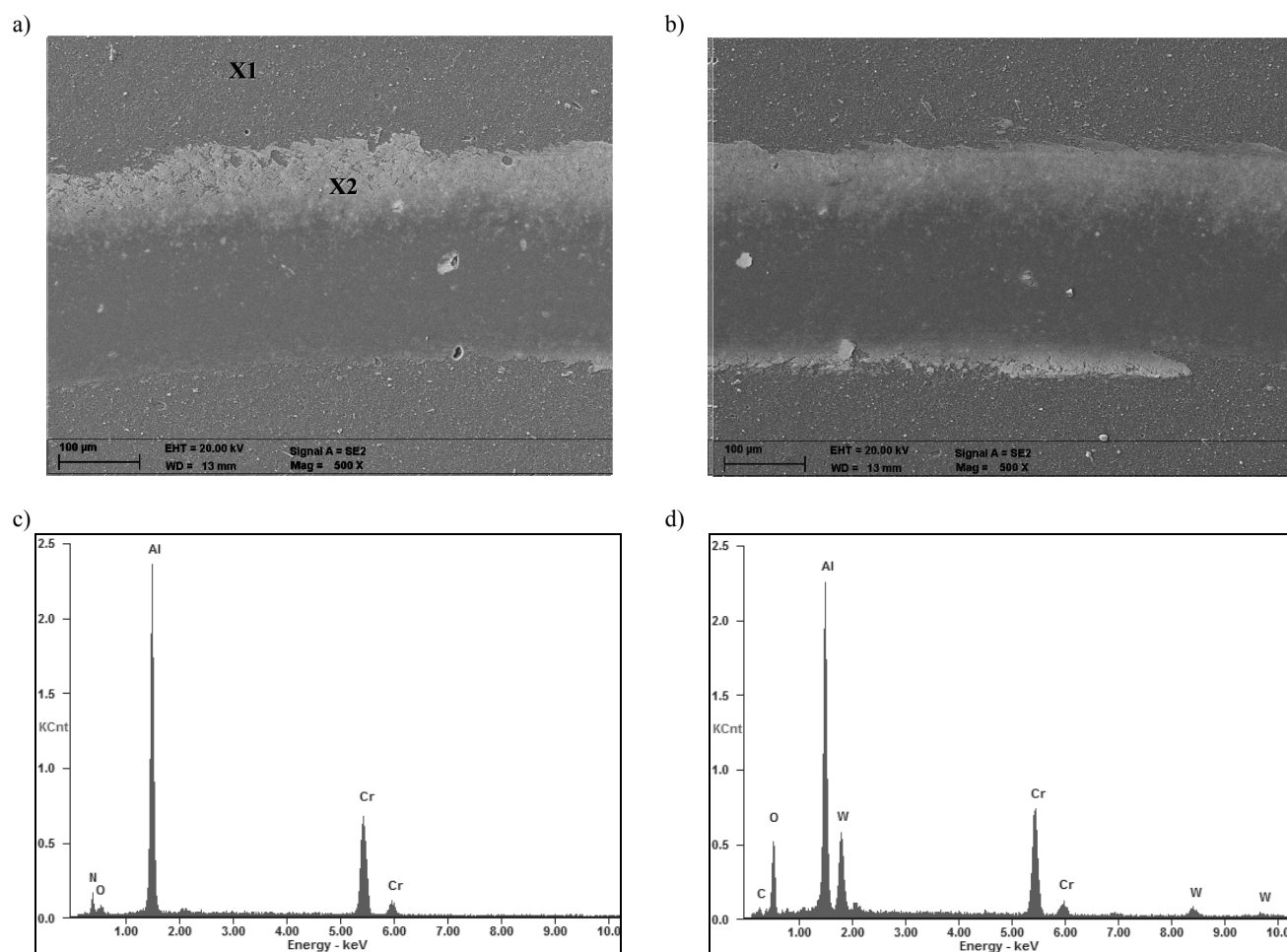


Fig. 6. a),b) Trace of tribological damage on (Al,Cr)N coating surface on sialon ceramic base and energy diagrams of EDS from micro-area: c) X1, d) X2

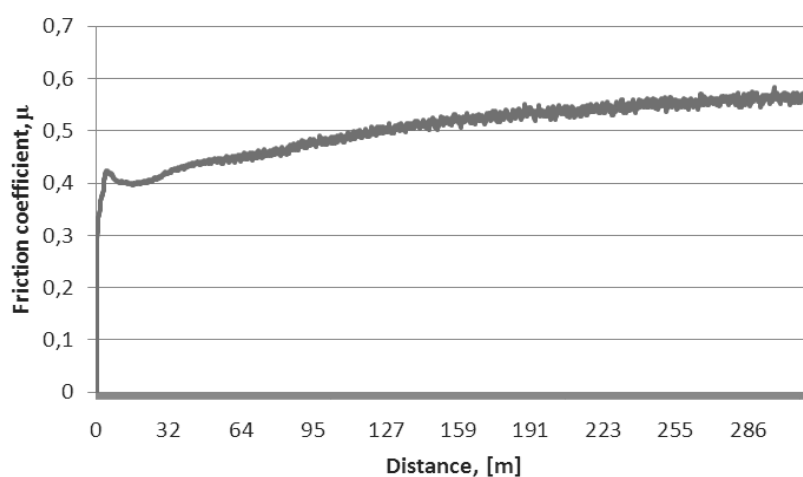


Fig. 7. Friction coefficient diagram depending on friction path during the „pin-on-disc” test for (Al,Cr)N coating laid on sialon ceramic base



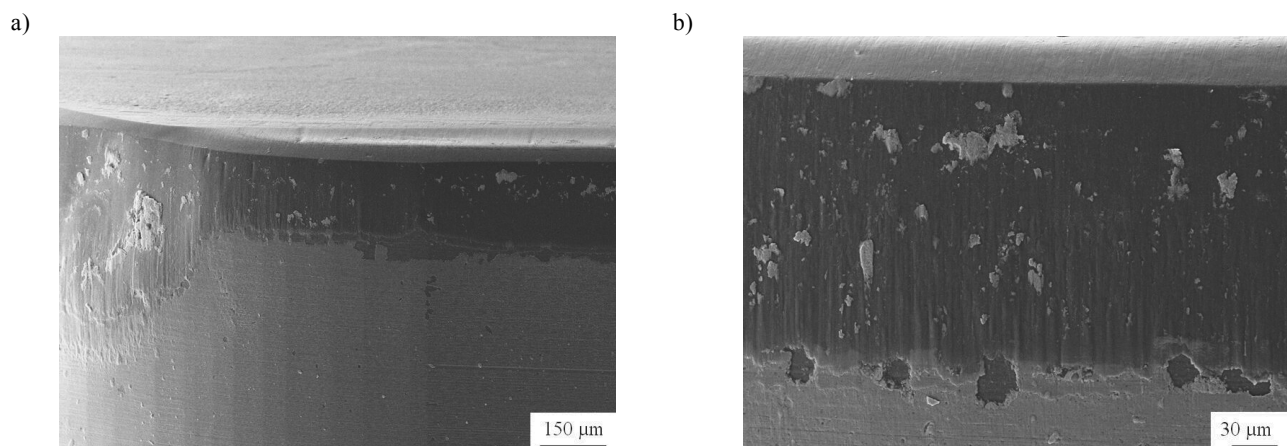


Fig. 8. Width of the VB tool flank for sialon tool ceramics with: a) Ti(B,N) coating - after 5 minutes of machining and b) (Ti,Zr)N coating -after 5.5 minutes of machining

#### 4. Summary

The application of gradient and multi-component coatings, most of all (Al,Cr)N and (Al,Cr)N+(Ti,Al)N laid on tool ceramics type SiAlON causes the growth of durability by up to 300% which, together with the possibility to use them in pro-ecological dry machining processes, without the use of coolants or lubricants, qualifies the coatings for wide range of industrial applications on machining tools. It was also found that the growing durability of the coated blades correlated with the coatings adherence to the base and growing micro-hardness of the coated plates, as compared to uncoated multi-blade plates.

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#### References

- [1] A.E. Reiter, V.H. Derflinger, B. Hanselmann, T. Bachmann, B. Sartory, Investigation of the properties of  $Al_{1-x}Cr_xN$  coatings prepared by cathodic arc evaporation, *Surface and Coatings Technology* 200 (2005) 2114-2122.
- [2] 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.
- [3] L.A. Dobrzański, D. Pakuła, Comparison of the structure and properties of the PVD and CVD coatings deposited on nitride tool ceramics, *Journal of Materials Processing Technology* 164-165 (2005) 832-842.
- [4] D. Pakuła, L.A. Dobrzański, K. Gołombek, M. Pancielejko, A. Křiž, Structure and properties of the  $Si_3N_4$  nitride ceramics with hard wear resistant coatings, *Journal of Materials Processing Technology* 157-158 (2004) 388-393.
- [5] D. Pakuła, M. Staszuk, Multilayer and multicomponent PVD coatings deposited on the ceramic tool materials, *Proceedings of the 10<sup>th</sup> Conference on "Coatings and Layers"*, Rožnov pod Radhoštěm, 2011, 129-134.
- [6] A.E. Reiter, V.H. Derflinger, B. Hanselmann, T. Bachmann, B. Sartory, Investigation of the properties of  $Al_{1-x}Cr_xN$  coatings prepared by cathodic arc evaporation, *Surface and Coatings Technology* 200 (2005) 2114-2122.
- [7] B.A. Movchan, K.Yu. Yakovchuk, Graded thermal barrier coatings, deposited by EB-PVD, *Surface and Coatings Technology* 188-189 (2004) 85-92.
- [8] D. Pakuła, L.A. Dobrzański, A. Křiž, M. Staszuk, Investigation of PVD coatings deposited on the  $Si_3N_4$  and sialon tool ceramics, *Archives of Materials Science and Engineering* 46/1 (2010) 53-60.
- [9] D. Pakuła, L.A. Dobrzański, K. Gołombek, M. Pancielejko, A. Křiž, Structure and properties of the  $Si_3N_4$  nitride ceramics with hard wear resistant coatings, *Journal of Materials Processing Technology* 157-158 (2004) 388-393.
- [10] L.A. Dobrzański, D. Pakuła, E. Hajduczek, Structure and properties of the multi-component TiAlSiN coatings obtained in the PVD process in the nitride tool ceramics, *Journal of Materials Processing Technology* 157-158 (2004) 331-340.
- [11] L.A. Dobrzański, D. Pakuła, A. Křiž, M. Soković, J. Kopač, Tribological properties of the PVD and CVD coatings deposited onto the nitride tool ceramics, *Journal of Materials Processing Technology* 175 (2006) 179-185.
- [12] L.A. Dobrzański, D. Pakuła, Comparison of the structure and properties of the PVD and CVD coatings deposited on nitride tool ceramics, *Journal of Materials Processing Technology* 164-165 (2005) 832-842.
- [13] 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.

- [14] L.A. Dobrzański, M. Staszuk, K. Gołombek, A. Śliwa, M. Pancielejko, Structure and properties PVD and CVD coatings deposited onto edges of sintered cutting tools, *Archives of Metallurgy and Materials* 55/1 (2010) 187-193.
- [15] L.A. Dobrzański, M. Staszuk, PVD and CVD gradient coatings on sintered carbides and sialon tool ceramics, *Journal of Achievements in Materials and Manufacturing Engineering* 43/2 (2010) 552-576.
- [16] M. Betiuk, T. Borowski, K. Burdyński, The (Ti,Al)N, (Ti,Al)C and (Ti,Al)CN multicomponent coatings synthesis in low pressure of DC arc discharge, *Engineering Materials* 6 (2008) 674-678 (in Polish).
- [17] M. Łapa D. Batory, Improving adhesion and wear resistance of carbon coatings using TiC gradient layers, *Journal of Achievements in Materials and Manufacturing Engineering* 20 (2007) 415-418.
- [18] M. Soković, J. Kopač, L.A. Dobrzański, J. Mikula, K. Gołombek, D. Pakuła, Cutting characteristics of PVD and CVD - Coated ceramic tool inserts, *Tribology in Industry* 28/1-2 (2006) 3-8.
- [19] M. Staszuk, The structure and properties of PVD and CVD gradient coatings deposited on sialons and sintered carbides. PhD Thesis, Silesian University of Technology Library, Gliwice, 2009 (in Polish).
- [20] M. Wysiecki, *Contemporary Tool Materials*, WNT, Warsaw, 1997, (in Polish).
- [21] S. PalDey, S.C. Deevi, Properties of single layer and gradient (Ti,Al)N coatings, *Materials Science and Engineering A361* (2003) 1-8.
- [22] Shuichi Kawno, Junichi Takahashi, Shiro Shimada, The preparation and spark plasma sintering of silicon nitride-based materials coated with nano-sized TiN, *Journal of the European Ceramic Society* 24 (2004) 309-312.
- [23] T. Liu, C. Dong, S. Wu, K. Tang, J. Wang, J. Jia, TiN gradient and Ti/TiN multi-layer protective coatings on Uranium, *Surface and Coating Technology* 201 (2007) 6737-6741.
- [24] T. Wierchoń, Structure and properties of multicomponent and composite layers produced by combined surface engineering methods, *Surface and Coatings Technology* 180-181 (2004) 458-464.
- [25] Yin-Yu Chang, Da-Yung Wang, Chi-Yung Hung, Structural and mechanical properties of nanolayered TiAlN/CrN coatings synthesized by a cathodic arc deposition process, *Surface and Coatings Technology* 200 (2005) 1702-1708.
- [26] L. Cunha, et al, Effect of thermal treatments on the structure of MoN<sub>x</sub>O<sub>y</sub> thin films, *Vacuum* 82/12 (2008) 1428-1432.
- [27] D. Pakuła, Structure and properties of multicomponent coatings deposited onto sialon tool ceramics, *Archives of Materials Science and Engineering* 52/1 (2011) 54-60.
- [28] 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.
- [29] L.A. Dobrzański, D. Pakuła, J. Mikula, K. Gołombek, Investigation of the structure and properties of coatings deposited on ceramic tool materials, *International Journal of Surface Science and Engineering* 1/1 (2007) 111-124.
- [30] M. Pancielejko, W. Precht, A. Czyżniewski, Tribological properties of PVD titanium carbides, *Vacuum* 53 (1999) 57-60.
- [31] W. Precht, M. Pancielejko, A. Czyżniewski, Structure and tribological properties of carbon and carbon nitride films, obtained by the ARC method, *Vacuum* 53 (1999) 109-112.
- [32] M. Pancielejko, A. Czyżniewski, V. Zavaleyev, A. Pander, K. Wojtalik, Optimization of the deposition parameters of DLC coatings with the MCVA method, *Archives of Materials Science and Engineering*, 54/2 (2012) 60-67.
- [33] A.D. Dobrzańska-Danikiewicz, K. Gołombek, D. Pakuła, J. Mikula, M. Staszuk, L.W. Żukowska, Long-term development directions of PVD/CVD coatings deposited onto sintered tool materials, *Archives of Materials Science and Engineering* 49/2 (2011) 69-96.