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Investigation of PVD coatings deposited on the Si_3N_4 and sialon tool ceramics

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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 Si_3N_4 and sialon tool ceramics. The Ti(B,N), Ti(C,N), (Ti,Zr)N and (Ti,Al)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 and using the X-ray diffractometer. 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.

Findings: Deposition of the multicomponent gradient coatings with the PVD method, based on the B, Al and Zr solid secondary solution in the TiN titanium nitride, isomorphous with the alternating pure titanium nitride TiN, on tools made from nitride ceramics and sialon's ceramics, results in the increase of mechanical properties in comparison with uncoated tool materials, deciding thus the improvement of their working properties.

Research limitations/implications: Ti(B,N), Ti(C,N), (Ti,Zr)N and (Ti,Al)N multicomponent and gradient coatings can be applied for cutting ceramic tools.

Originality/value: Comparison of the wide range of modern sintered tool materials with wide unique set of PVD coatings.

Keywords: Thin & thick coatings; PVD; Si_3N_4; SIALON

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

Service properties of products and their constituent elements depend to a big extent on structure and properties of their surface layers. The properly selected substrate and surface layers materials and processes of forming their structure and properties ensure the required service properties at the relatively low costs. The strive to produce better, more modern, and first of all most efficient tools induces to improve the technologies implemented to date or to search new solutions to attain more advantageous service properties of the manufactured elements. The nisus for attaining the specific properties like limiting the tribilogical wear is the aim and research object for many engineers worldwide. Composition of the surface layers, that can be modified – among others – by deposition of coatings from the gaseous phase, has the significant effect on those properties (employment of the PVD- Physical Vapour Deposition and CVD- Chemical Vapour Deposition brings measurable for more than half of a century now) [1-15].

Technologies of the chemical and physical deposition of layers from the gaseous phase make development of coatings possible whose structure and properties are precisely defined by the relevant selection of elements constituting the coating deposited. In this way the hard abrasion wear resistant coatings are made, of great significance in development of many industry branches, as elements covered with such coatinge are characteristic of the enormous growth of their hardness and abrasion wear resistance compared to elements that are not coated with the protection layer. Deposition of the hard layer of nitrides, carbides, or oxides onto the material surface, among others in the PVD process is one of the most intensely developing branches for improving the service properties of the functional elements. Hard metal nitrides coatings extend the life of elements coated with these layers, increasing their abrasion wear resistance and their resistance to the aggressive chemical environment action [16-40].

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

2. Experimental procedure

The investigations were carried out on the multi-point inserts made from the Si_3N_4 nitride ceramics and sialon tool ceramics uncoated and coated in the PVD process with thin coatings. The inserts were coated in the PVD process – Cathodic Arc Evaporation (CAE) Ti(B,N), Ti(C,N), (Ti,Zr)N and (Ti,Al)N coating. Specifications of the investigated materials are presented in Table 1.

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 Ra roughness parameter of the uncoated and coated surfaces was determined on the RankTaylor Hobson Surftec 3+ device with the measurement length of 0.25 mm. The measurements were made in two orthogonal directions with the measurement accuracy of 0.01 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.

Adhesion evaluation of the coatings on the investigated inserts was made using the scratch test on the CSEM REVETEST device, by moving the diamond penetrator along the examined specimen's surface with the gradually increasing load. The tests were made with the following parameters: load range 0-100 N, load increase rate (dL/dt) 100 N/min, penetrator's travel speed (dx/dt) 10 mm/min, acoustic emission detector's sensitivity AE 1. The critical load LC, at which coatings' adhesion is lost, was determined basing on the registered values of the acoustic emission AE.

Phase composition analyses of investigated samples were made on the PANalytical X'Pert PRO diffractometer, working in goniometer system (using the filtered X-ray Co Kα, step 0.05, time of counting 10 sec.) at the voltage of 40 kV and tube current of 30 mA.

Observations of the investigated coatings' structures were carried out on the transverse fractures on the scanning electron microscope (SEM) Zeiss Supra 35. 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], maximum magnifications are 10000x. The specimens with notches cut on them were cooled in liquid nitrogen. Topography of the investigated coatings' surfaces was also examined on the scanning electron microscope.

Table 1.
Characteristics of the PVD coatings deposited on the Si_3N_4 and sialon tool ceramics

Substrate	Coating	Coating thickness, μm	Roughness Ra, μm	Microhardness HV 0.05	Critical load Lc, N
Si_3N_4 ceramics	uncoated	-	0.06	1886 *	-
	Ti(B,N)	2.1	0.26	2898	12
	Ti(C,N)	2.1	0.34	3188	14
	(Ti,Zr)N	1.7	0.29	2798	30
	(Ti,Al)N	0.7	0.23	3408	42
sialon tool ceramics	uncoated	-	0.06	2035 *	-
	Ti(B,N)	1.3	0.25	2676	13
	Ti(C,N)	1.5	0.23	2872	25
	(Ti,Zr)N	2.3	0.40	2916	21
	(Ti,Al)N	5.0	0.28	2961	21

* - HV 0.3

3. Discussion of the experimental results

It was found out in roughness testing that the lowest average roughness is characteristic for the uncoated inserts, whose roughness was 0.06 μm for Si_3N_4 ceramics and 0.06 μm for sialon ceramics. The average roughness value of the analysed inserts with the anti-wear coatings is in the 0.23-0.40 μm range. Sialon insert with the (Ti,Zr)N coating had the highest roughness of all tested specimens. The lowest average roughness value were attained by the test piece with the (Ti,Al)N layer on Si_3N_4 substrate and Ti(C,N) layer on sialon substrate (Table 1).

It was found out in hardness testing that deposition of the anti-wear coatings onto the inserts from the nitride ceramics makes it possible to increase their hardness even by 80% (insert with the (Ti,Al)N coating achieved the average hardness of 3408 HV 0.05) compared to the uncoated insert from the nitride ceramics, whose hardness is the 1886 HV 0.3. The lowest hardness from all analysed inserts with the anti-wear coatings has the insert with the (Ti,Zr)N coating (2798 HV 0.05), yet hardness of this insert is higher by more than 50% than hardness of the uncoated insert. The other investigated specimens achieved hardness of 2898 HV 0.05 – insert with the Ti(B,N) coating, and 3188 HV 0.05 – insert with the Ti(C,N) coating. The results of microhardness tests for coatings deposited on both investigating substrates as well as uncoated substrates were presented in Table 1. The microhardness of sialon substrates is equal 2035 HV 0.05 and in each case increase after deposited a coating. Microhardness of investigated coatings is included in a range from 2676 HV0.05 to 2961 HV0.05. It was found that a (Ti,Al)N coating shows higher hardness on substrate from sialon ceramics.

It was found out during adhesion tests of coatings deposited onto the ceramics substrates that spalling and delamination are the most common forms of their defects (Figs. 3, 4). The (Ti,Al)N coating on Si_3N_4 ceramics remained without damages for the longest time from all analysed layers and it was necessary to increase load twofold to make the first spallings appear. Damage of this coating was of the cyclic delamination character at first on both scratch edges and two-sided spallings were observed along with the load increase. Total coating delamination occurred at the load order of magnitude of 90 N. The Ti(B,N) coating revealed only single damages in the form of delamination on the edge and inside of the scratch after applying the load. These damages were getting more concentrated on the scratch surface along with increasing the load and one could observe partial and band delaminations, as well as single side and two-side spalling. At the critical load the total coating delamination occurred (Figs. 2, 3 Table 1).

The Ti(C,N) coating was subjected to the total delamination soonest. Spalling and band delamination occurred already at the beginning of the scratch, and the total coating delamination occurred at the load of only 13 N deposited on Si_3N_4 ceramics and 25 N on sialon ceramics (Fig. 3b).

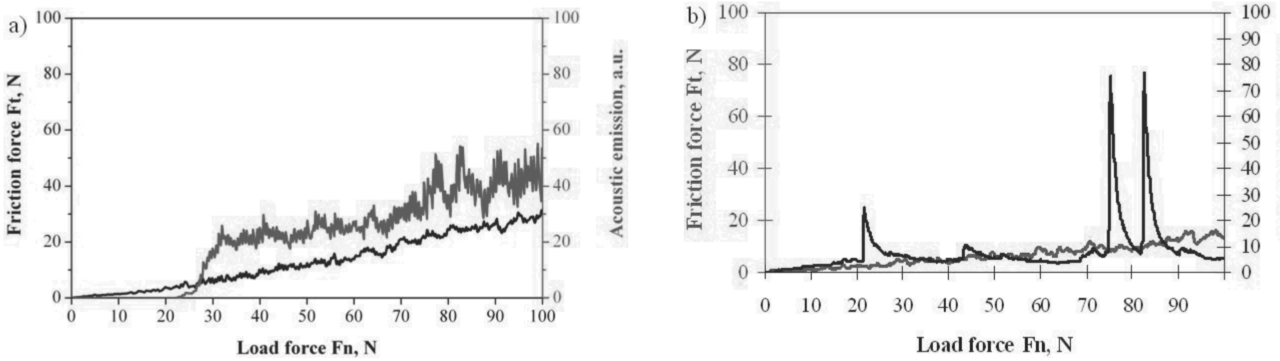


Fig. 1. Scratch test: Acoustic emission (AE) and friction force Ft as a function of the load force Fn for a) Ti(C,N), b) (Ti,Zr)N coating surface deposited onto the sialon ceramics substrate

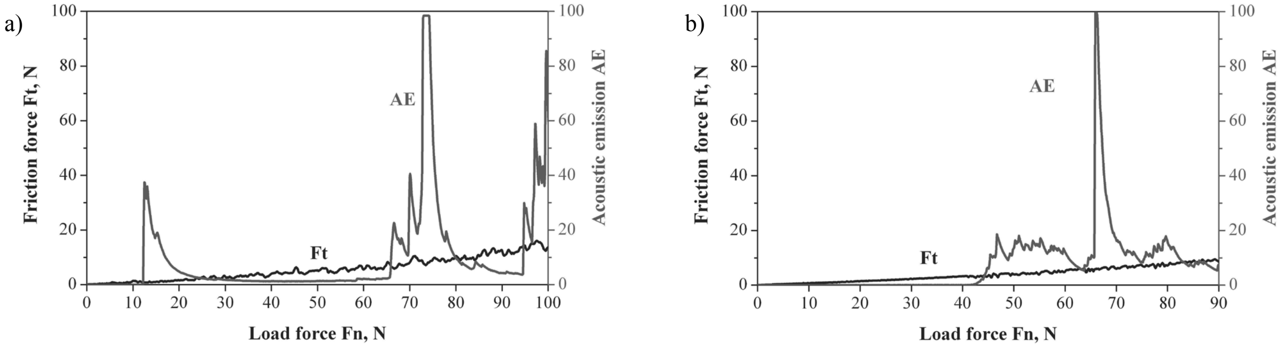


Fig. 2. Scratch test: Acoustic emission (AE) and friction force Ft as a function of the load force Fn for a) Ti(B,N), b) (Ti,Al)N coating surface deposited onto the Si_3N_4 ceramics substrate

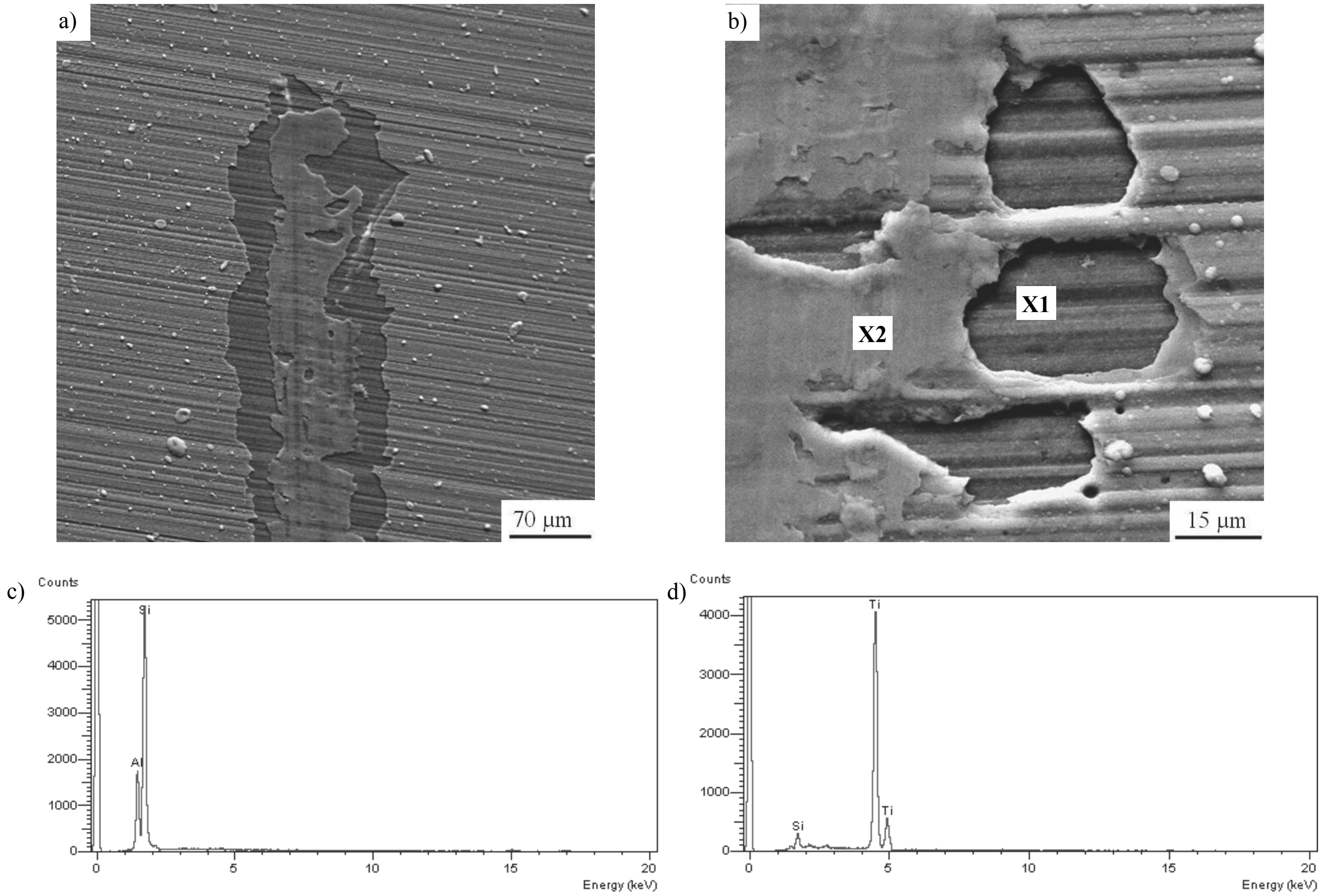


Fig. 3. Characteristic failure of the a) Ti(B,N) and b) Ti(C,N) coatings deposited on sialon tool ceramics developed during the adhesion scratch test, c) X-ray energy dispersive plot the area X1 as in a figure b, d) X-ray energy dispersive plot the area X2 as in a figure b

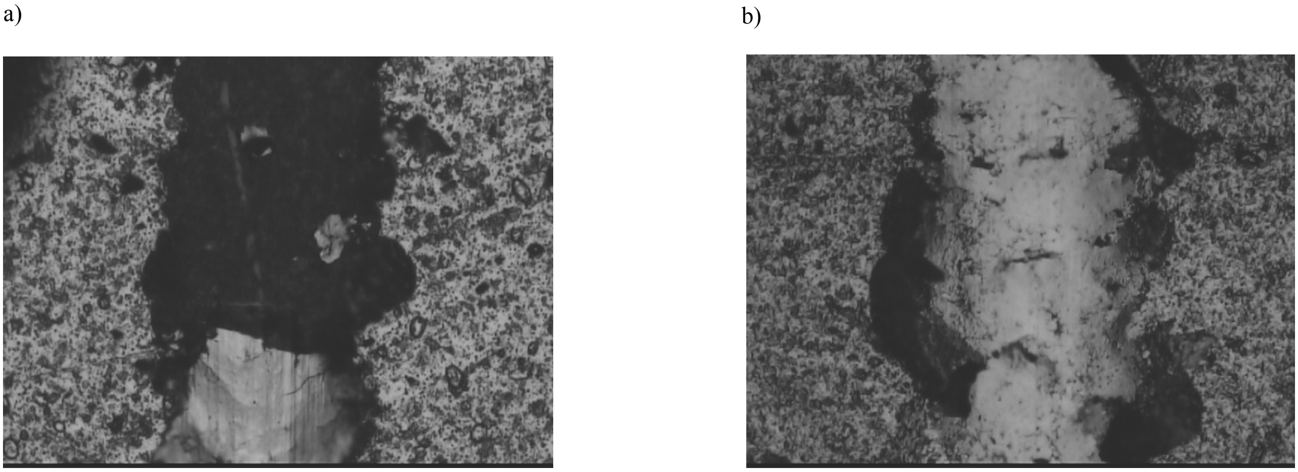


Fig. 4. Characteristic failure of the a) (Ti,Zr)N and b) (Ti,Al)N coatings deposited on Si_3N_4 tool ceramics developed during the adhesion scratch test

Adhesion of the (Ti,Zr)N layer is not good either. The edge delamination occurred at the slight load and inside of the scratch and next the single sided spallings occurred and at the critical load of more than 30 N the total delamination happened (Fig. 4).

Adhesion of this same coatings deposited onto sialon tool ceramics are usually lower than deposited onto Si_3N_4 ceramics. Values in this case are equal between 13 and 25 N (Table 1).

It was found based on results obtained from the X-ray phase qualitative analysis that the most intensive reflexes coming from the substrates are those coming from (200), (101), (210), and (321) planes. Diffraction analysis of XRD researches that structure of sialons is consisted of β-Si_3N_4 phase.

Moreover, occurrence of the TiN phase was revealed in three out of four coatings deposited onto the both substrates, whose most intensive reflexes come from the (111), (200), and (220) planes. In the Ti(C,N) coating (as the only one) no TiN phase mentioned above was detected, however the Ti(C,N) phase was found in it (Fig. 5).

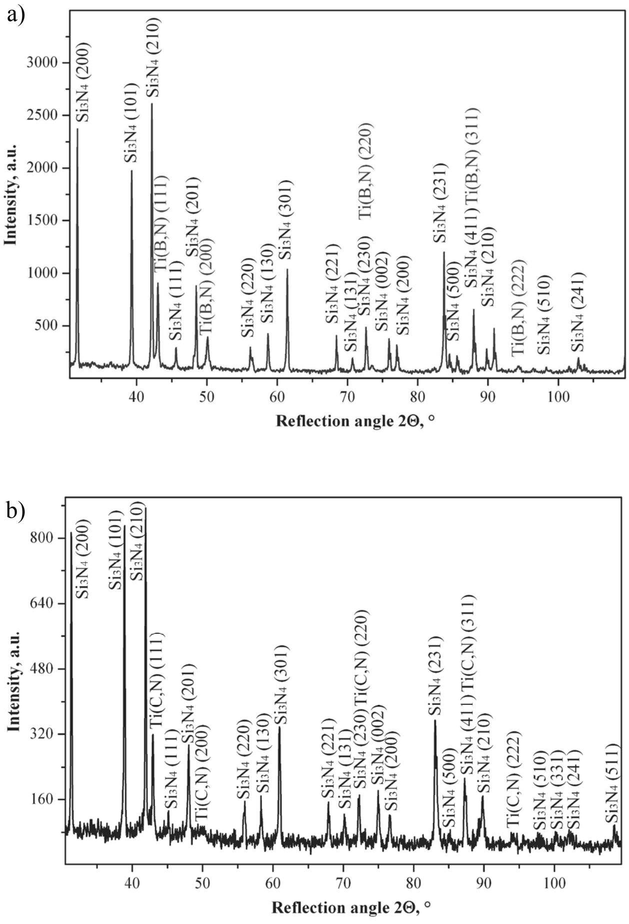


Fig. 5. X-ray diffraction pattern of a) Ti(B,N) coating deposited on Si_3N_4 ceramics, b) Ti(C,N) coating deposited on sialon tool ceramics

In case of coatings deposited by the physical deposition from the gaseous phase, distinction of the TiN phase from the Ti(B,N), (Ti,Zr)N or (Ti,Al)N ones is impossible using the diffraction method due to the isomorphism of these phases.

It was found in the metallographic examinations, for which the scanning electron microscope was used, that the multicomponent coatings deposited onto the inserts from the Si_3N_4 nitride ceramics and sialon tool ceramics are characteristic of the tight adhesion to the substrate. One can observe in the fracture photos the nearly uniform coatings deposition and their compact structure (Fig. 6).

Surface topography of the deposited layers demonstrates inhomegeneity in the form of drops which is characteristic for the cathode CAE evaporation (Fig. 7). All these inhomogeneity observed in SEM cause an increase of roughness surface in comparison with uncoated inserts.

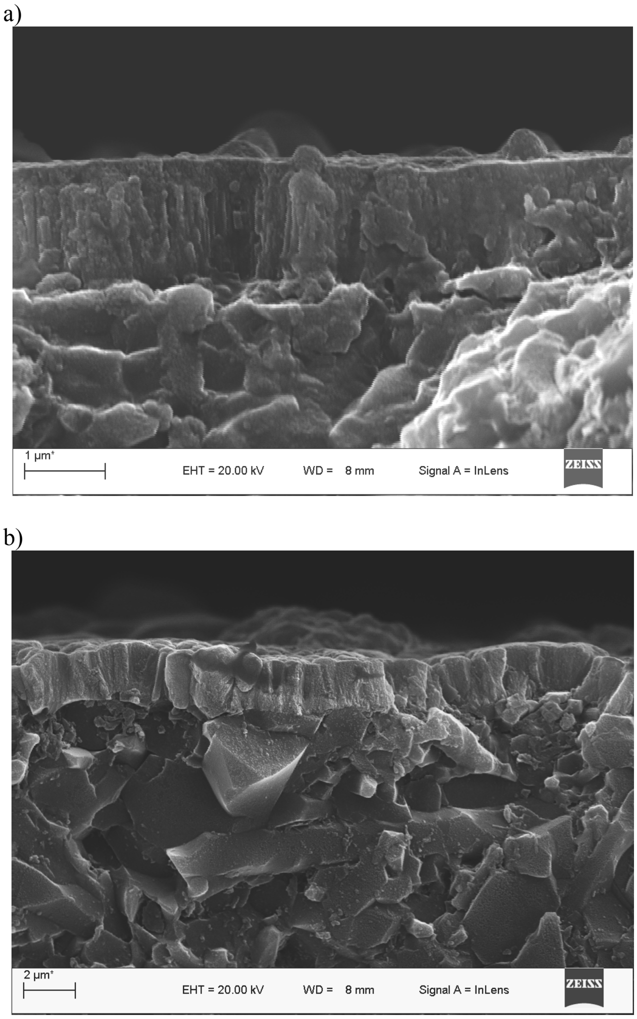


Fig. 6. Fracture surface of the a) (Ti,Zr)N and b) Ti(C,N) coatings

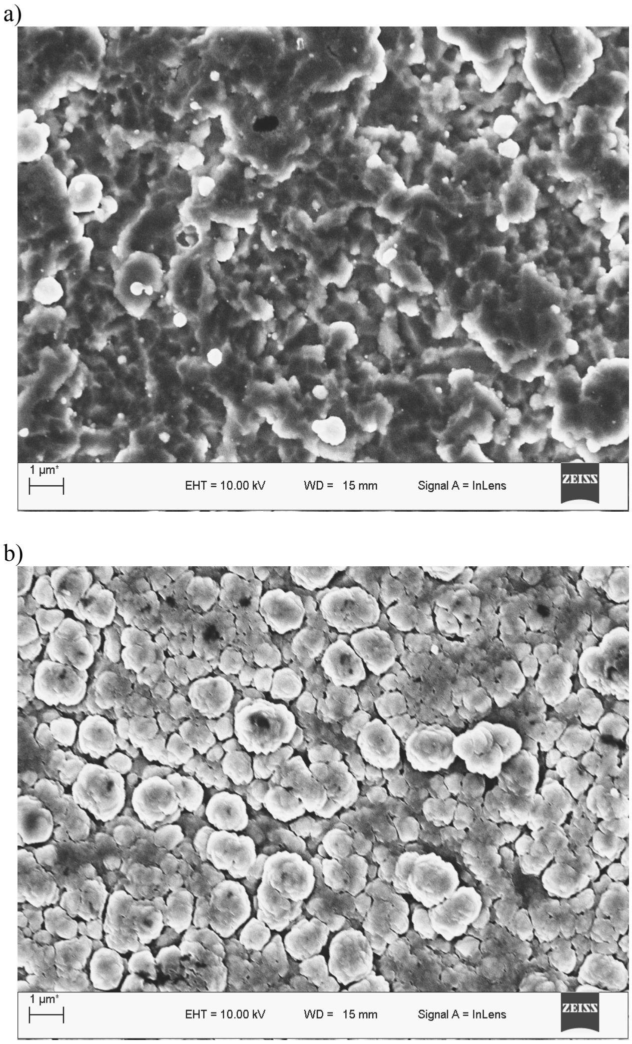
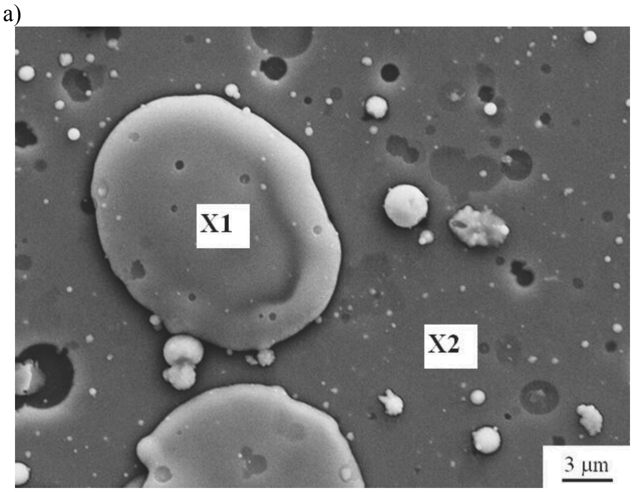


Fig. 7. Topography of the a) Ti(B,N) and b) (Ti,Al)N coating surface deposited on Si_3N_4 substrate



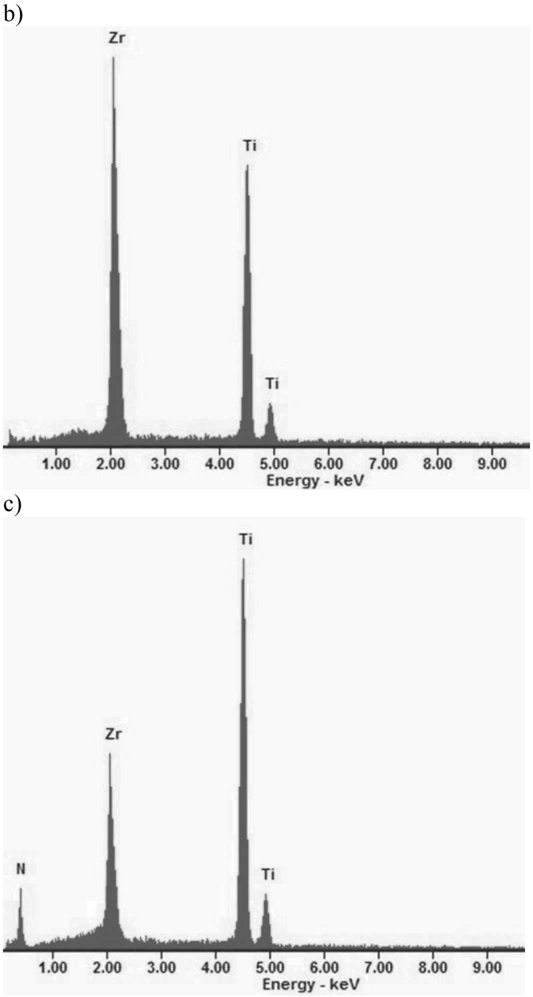


Fig. 8. Topography of the (Ti,Zr)N coating surface deposited on sialon substrate, b) X-ray energy dispersive plot the area X1 as in a figure a, c) X-ray energy dispersive plot the area X2 as in a figure a

The X-ray chemical composition microanalysis revealed occurrences of the anticipated elements coming from the substrate or constituting the particular coatings, i.e., nitrogen, titanium, zirconium, and aluminium (Fig. 8).

4. Conclusions

The following conclusions were drawn based on the tests carried out:

1. The coatings were deposited uniformly onto the Si_3N_4 ceramics and sialon tool ceramics substrates, are characteristic of the compact structure and tight adhesion, albeit one can observe occurences of drops from the evaporated disk on their

surface, which is common for coatings deposited with the PVD methods.
2. Depositing the multicomponent anti-wear coatings onto the Si_3N_4 nitride ceramics and sialon ceramisc makes hardness growth possible even by 80% (the (Ti,Al)N layer on Si_3N_4 attained the average hardness of 3408 HV 0.05).
3. Deposition process type (CAE) and the substrate from the nitride and sialon ceramics, whose polarisation makes satisfactory adhesion of the coatings deposited to the substrate surface impossible, have an effect on the poor adhesion of the analysed coatings.
4. High hardness and adhesion of the (Ti,Al)N coating (in comparison with the other investigated layers) suggest that it makes life extension of the element possible, on which it is deposited increasing its hardness and wear resistance.

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