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STRUCTURE AND PROPERTIES OF THE TOOL CERAMICS WITH HARD WEAR RESISTANT COATINGS

This work presents studies of the structure and functional properties of coatings deposited onto indexable inserts made of nitride and sialon tool ceramics with the required properties, i.e. high adhesion, microhardness, high resistance to abrasive and diffusion wear in working conditions of high-performance cuttings tools. In the present paper the results of the investigations of the structure, texture, mechanical and functional properties of the Ti(C,N), (Ti,Al)N, Ti(C,N)+(Ti,Al)N coatings were presented. The 80% increase in the hardness of the coatings in comparison to the substrate material was reported. Test coatings are characterized by good adhesion to the substrate. The maximum Lc load of (Ti,Al)N coat applied to the substrate from the nitride ceramics is equal to 42 N. In the studied coatings compressive stresses were found. The results of mechanical properties investigations, especially tribological ones correlate with the results of exploitation tests carried out during the cutting test.

Keywords: PVD coatings, Nitride Tool Ceramics, Sialon Tool Ceramics

1. Introduction

The application of the physical vapour deposition (PVD) technique for the surface treatment of indexable inserts made of nitride and sialon tool ceramics to achieve coatings with high resistance to abrasive wear allows to improve the properties of such materials in machining conditions. The optimum properties are achieved by increasing microhardness, reducing a friction factor and improving tribological contact conditions in the machined item - tool interface area. A tool, through the deposition of PVD coatings, is also secured against adhesive, diffusion wear and oxidation. The occurring stresses have great significance for the functional properties of hard antiwear coatings deposited onto the cutting edges of cutting tools. The stresses can be reduced after the complete PVD process by relaxing annealing. The temperature of the heat treatment should be higher than the temperature of deposition [1-3]. It is more beneficial when compressive stresses do exist in coatings, improving the mechanical properties of the coatings, such as microhardness and adhesion. Moreover, as a result of the considerable compressive stresses existing in the coating, a heated substrate in a machining process does not lead to the formation of cracks in the coating, but is reducing the value of compressive stresses in the coating, even by eliminating them completely. An insightful analysis of such properties allows to determine an optimum area of application of substrate materials made of tool ceramics as well as the investigated coatings obtained in PVD processes. It is very important to determine a correlation between the results of investigations of the structure, internal stresses and mechanical properties in relation to the wear determined as a result of abrasive resistance tests made using “pin on disc“ method and as a result of a technological cutting test [1-18].

The aim of the paper is the investigation of structure and properties of the PVD deposited coatings onto a substrate made of silicon nitride and sialon ceramics.

2. Materials

The research was carried out on multi-point inserts made of sialon and silicon nitride tool ceramics, non-coated and

TABLE 1

Specifications of the PVD coatings deposited on the tool ceramics

Substrate	Coating type	Coating composition	Coating thickness, mm
Nitride tool ceramics Si_3N_4	mono layer	Ti(C,N)	2.1
	gradient	(Ti,Al)N	0.7
	double layer	Ti(C,N)+(Ti,Al)N	2.0
Sialon tool ceramics SiAlON	mono layer	Ti(C,N)	1.8
	gradient	(Ti,Al)N	5.0
	double layer	Ti(C,N)+(Ti,Al)N	1.4

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coated with PVD coatings. The inserts were coated using the cathode arc evaporation process (CAE-PVD) with Ti(C,N), (Ti,Al)N, and Ti(C,N)+(Ti,Al)N coatings (Table 1).

3. Methodology

A scanning electron microscope Zeiss Supra 35 was used to observe both the structure and morphology of the obtained coatings, as well as damages to the coatings occurred after the examination of coating adhesion to the substrate and the cutting trials inserts.

Changes of the chemical concentration of the coating components in the direction perpendicular to the coating surface and concentration variations in the transition zone between the coating and the substrate material were evaluated based on tests with a GDOES 850A QDP glow discharge optical spectrometer by Leco Instruments. Due to the fact that substrates are non-conductive the investigations were performed in rf (Radio Frequency) mode.

The texture of the investigated coatings deposited onto the substrate made of sialon ceramics was were evaluated with an X´Pert PRO X-ray instrument by Panalytical. Pole figures were measured with the reflection method using an Euler's disc with the diameter of 187 mm and the specimen inclination angles of 0 to 75° in order to determine the distribution of normals for the selected plane and to determine Orientation Distribution Functions (ODFs) of the coatings obtained in PVD processes. An ODF analysis of the investigated materials with procedures available in LaboTex 3.0 software using the discreet ADC method with iteration operator was made [20].

The internal macro-stresses (measured in two perpendicular directions) of the examined PVD coatings with the $sin_2\psi$ method using X'Pert Stress Plus software incorporating data, as a database, necessary to calculate the values of material constants were determined [20].

The microhardness tests of the obtained coatings using SHIMADZU DUH 202 ultra microhardness tester were made. Measurements under the load of 0.07 N, eliminating influence of the substrate on the measurement results were made.

Adhesion evaluation of the coatings on the investigated inserts using the scratch test on the CSM REVETEST device, by moving the diamond penetrator along the examined specimen's surface with the gradually increasing load was made. The tests under 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 were made. The critical load LC, at which coatings' adhesion is lost, was determined basing on the registered values of the acoustic emission AE.

Tribological dry tests were carried out on the CSM „pin-on-disk" tester under the following conditions: counter-specimen – ball made from the WC titanium carbide with the 6 mm diameter, counter-specimen load – 5 N, friction radius – 5 mm, linear velocity – 0.1 m/sec, ambient temperature – 20C. Test was performed without lubricant.

Cutting ability of the investigated materials basing on the technological continuous cutting tests of the EN-GJL-250 grey cast iron with the hardness of about 215 HB was determined. The width VB = 0.30 mm 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 in the machining capability experiments were used: feed rate f = 0.2 mm/rev; depth of cut ap = 2mm; cutting speed v_c = 400m/min.

4. Results and discussion

On the basis of examinations undertaken using a scanning electron microscope (SEM) was confirmed that the coatings examined, having a column-like structure, were deposited evenly onto the substrate and adhere tightly. A Ti(C,N) coating is characterised by a compact structure without pores with column-like grains with the zone II acc. to Thornton's model (Fig. 1), whereas a graded coating (Ti,Al)N also features a column-like structure according to the zone I acc. to Thornton's model [21].

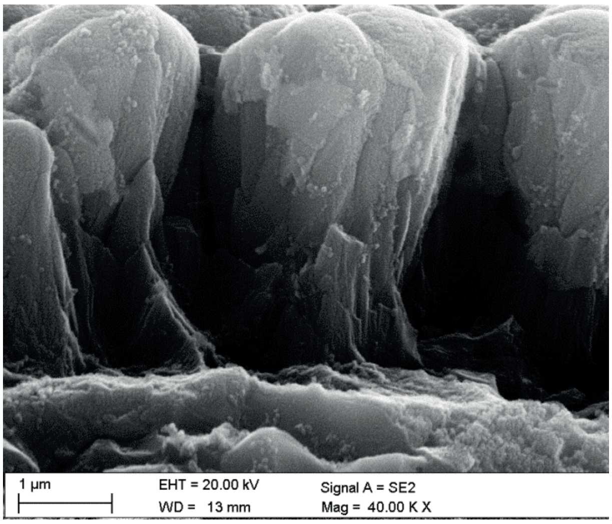
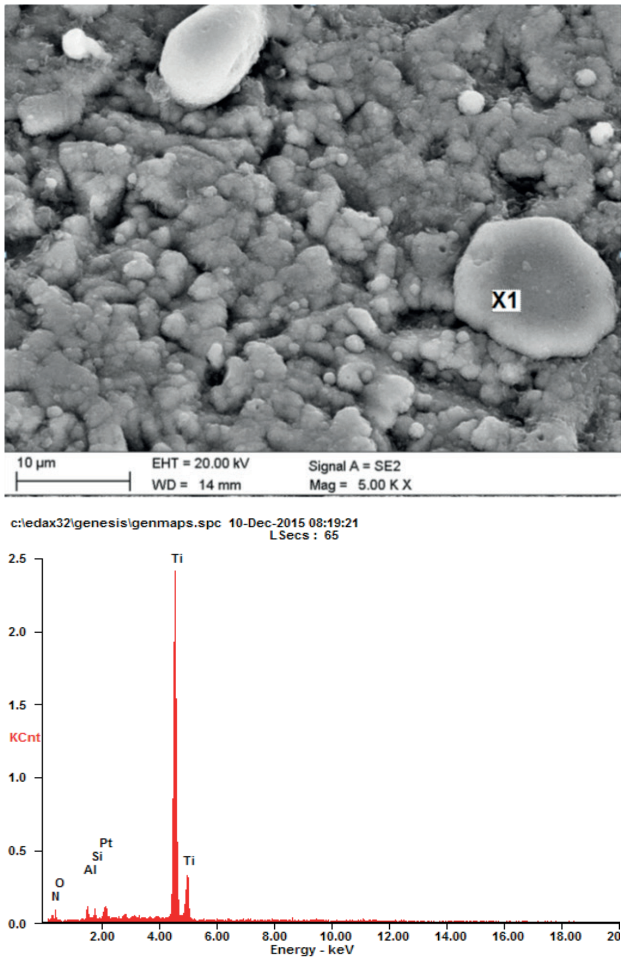


Fig. 1. Fracture surface of Ti(C,N) coatings

An inhomogeneity related to the occurrence of multiple droplet-shaped microparticles on the coating surface was found during the surface topography of the analysed PVD coatings observations, what is connected with the essence of the applied CAE process of coatings' deposition. The particles size varies and spans between the tenths of a micrometer to more than ten micrometers. The examinations of chemical composition of particles performed with an EDS X-ray scattered radiation energy spectrometer indicate that particles are formed from pristine metal - titanium, which is removed from a titanium target, and is deposited and solidified on the substrate surface (Fig. 2).

An analysis with a GDOES glow discharge optical spectrometer point out that the distribution of chemical elements in the examined micro-areas of the coating and substrate is correct. The elements forming part of the examined coatings are present exclusively in the coating area, and the elements forming part of nitride and sialon tool ceramics occur in the substrate area (Fig. 3). The examinations also reveal a rising concentration of elements forming part of the substrate in the zone between the substrate and the coating in the cases considered, accompanied by a decreasing concentration of the elements forming the coatings. This may confirm the existence of a very thin transition layer between the substrate material and the coating, improving the adhesion of the coatings deposited to the substrate.

a)



b)

Fig. 2. a) Surface topography and b) the X-ray energy dispersive plots from the microzones X1 of the Ti(C,N) coating surface, deposited onto the Si_3N_4 nitride ceramics

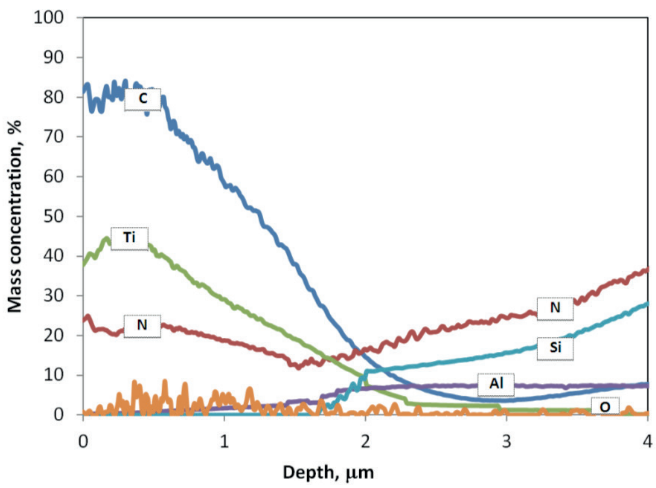
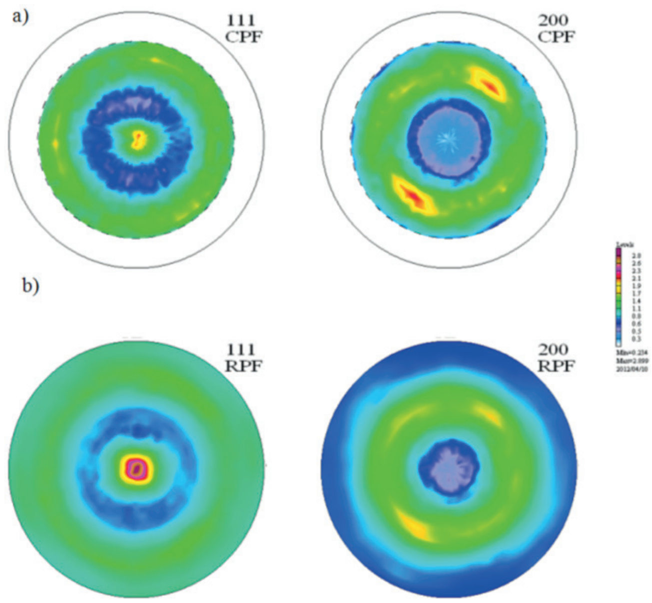


Fig. 3. Variations in the concentration of Ti(C,N) coating components and the substrate material from sialon ceramics analysed in GDOES spectrometer

Figures 4 and 5 show a texture of a Ti(C,N) coating created in a cathodic arc evaporation (CAE) process in the form of experimental pole figures designated as CPF, in the form of complete pole figures calculated with the ODF designated as RPF and as ODF. A texture analysis carried out with the reflection method allows to conclude, on the basis of single pole figures, that the distinguished growth plane in the Ti(C,N) coating is a plane from the {111} family with the fraction of the distinguished component of 12%. A qualitative texture analysis was only carried out for the (Ti,Al)N coating and for the double-layer Ti(C,N)+(Ti,Al)N coatings considering that the reflexes of the substrate material and of the coating coincide. The said analysis points out that the above coatings do not show a privileged growth orientation.

a)



b)

Fig. 4. Pole figures (111), (200) of Ti(C,N) coating obtained in the PVD process on sialon ceramics substrate: a) experimental, b) calculated from ODF

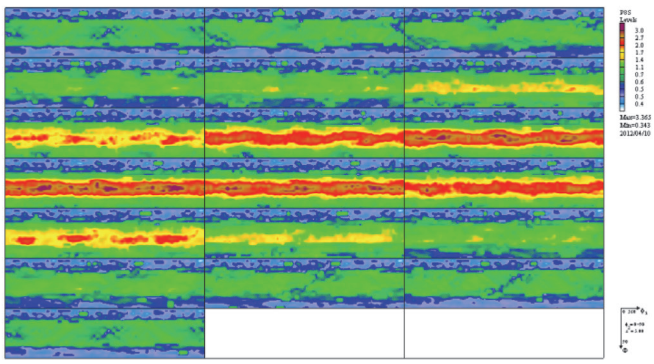


Fig. 5. Orientation distribution function of Ti(C,N) coating obtained in the PVD process on sialon ceramics substrate: a) cross section after φ2 (for the following φ2 values: 0, 5, 0…90º)

The presence of compressive stresses (Fig. 6) was identified based on the examinations of internal stresses of PVD coatings achieved on the Si_3N_4 and SiAlON substrate. The highest relative value of a compressive stress (σ_φ = 1912 MPa) was noticed for a Ti(C,N)+(Ti,Al)N coating deposited onto a sialon ceramics substrate, whilst the lowest relative value of a compressive stress (σ_φ = 921

MPa) was determined for a Ti(C,N) coating deposited onto a sialon ceramics substrate. It was also found that the growth of compressive stresses is having influence on enhanced microhardness as well as improved adhesion and wear resistance of the investigated coatings. Especially, in the case of coatings (Ti,Al)N and Ti(C,N)+(Ti,Al)N, which increased the durability of the cutting edge such an effect is observed. As a result of heating the substrate in the cutting process, probably there may be a reduction of compressive stresses occurring in the coating or even to their total elimination. The cutting temperature was higher than temperature of coating, what is close to conditions of stress relief annealing, approximating the material condition to the equilibrium conditions [1]. Furthermore, densification of the coatings structure caused by compressive stress occurred about relatively high absolute values is a factor influence on increase the microhardness of these coatings. [2,3] (Fig. 7 and 9).

The microhardness tests revealed that the uncoated ceramics tool material has hardness from 1850 HV in case of Si_3N_4 substrate to 2035 HV in case of SiAlON substrate. Deposition of the PVD coatings onto a substrate made of nitride and sialon ceramics based specimens causes hardness increase of the surface layer reaching from 2786 to 3408 HV, that is up to 80% more compared to the substrate hardness. The highest hardness of 3408 HV in case of the (Ti,Al)N coating deposited onto nitride ceramics was observed. No dependence was revealed between the hardness of deposited surface layer and the substrate hardness (Fig. 7).

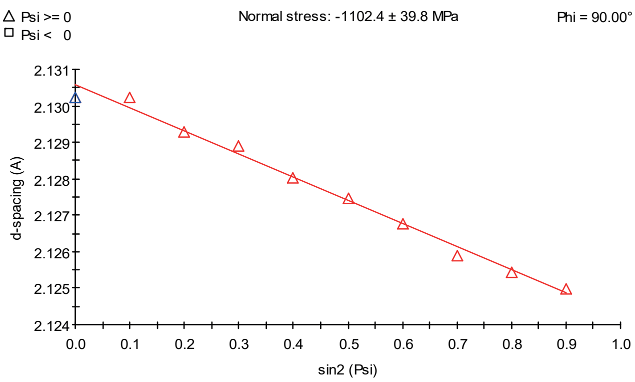


Fig. 6. Variations in the interplanar distance value of d reflex (200) of the Ti(C,N) coating obtained in PVD process on the substrate made of s ceramics in the function of $\sin^2\psi$ (measurement of stresses with the $\sin^2\psi$ method) - measurement along the main cutting edge

The values of the critical load L_c (AE), characterising the adhesion of the examined PVD coatings to a Si_3N_4 nitride ceramics and SiAlON sialon ceramics substrate, were determined with a scratch test, with a load growing in a linear manner. It was pointed out for the analysed coatings that the highest critical load value, $L_c = 42$ N, is exhibited by a (Ti,Al)N coating deposited onto a nitride ceramics substrate, whereas the lowest value, $L_c = 14$ N, by a Ti(C,N) coating also deposited onto a nitride ceramics substrate. The critical load Lc, for the coatings achieved on a sialon tool ceramics substrate, varies between 26 and 36 N (Fig. 9).

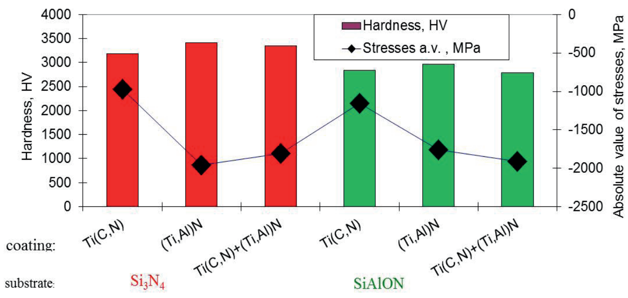


Fig. 7. Comparison of microhardness and residual stresses of investigated coatings deposited on the nitride and sialon ceramics

The damages created as a result of the scratch test of PVD coatings, especially double-layer coatings, deposited onto nitride ceramics and sialon ceramics, are characterised by a large number of single and double-sided coating cracks at the scratch peripheries. Partial delamination inside the scratch and at its end was also noticed. If a load is increased, this often causes conformal cracks and the periodical flaking of a coating (Fig 8). The smallest damages – singular delamination in the initial zone of the scratch, small one-sided chippings in the central zone of the scratch and double-sided chippings at the end of the scratch – were observed in (Ti,Al)N coatings, with the (Ti,Al)N coating deposited onto a nitride ceramics substrate having the best adhesion (Fig. 8).

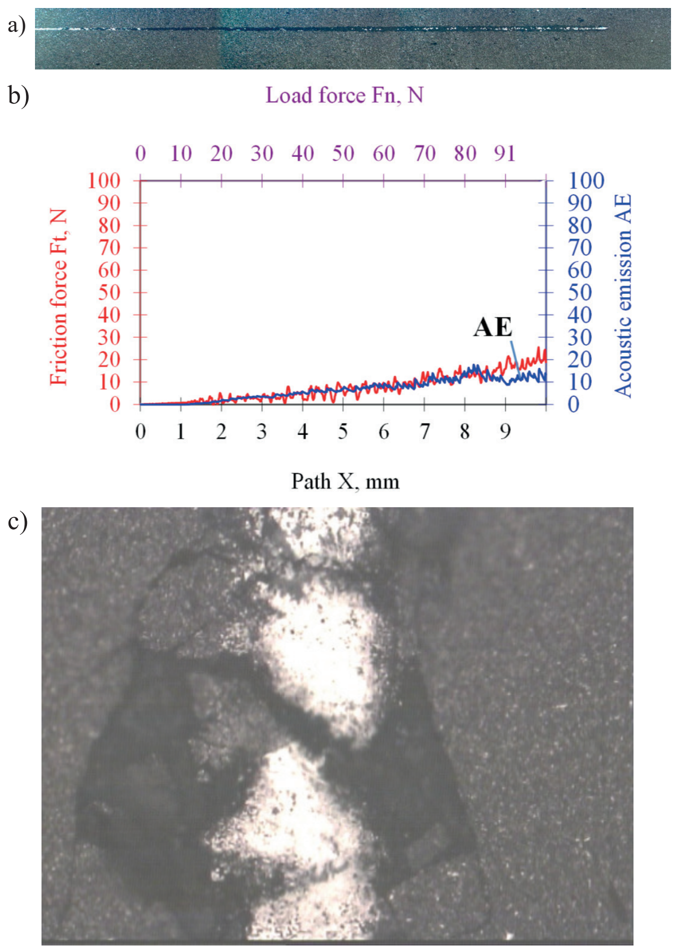


Fig. 8 (a) Trace of the scratch test; (b) diagram of the dependence of friction force (Ft) and Acoustic Emission (AE) on the load and (c) failure by 30 N load – for the Ti(C,N) coating deposited on Si_3N_4 nitride ceramics (magnification 200x)

An abrasive wear resistance test of the studied materials was made with the pin on disc method. The constant number of cycles, n=10,000, for each of the coatings, allowing to carry out a metallographic comparative analysis of the coating damage track, was assumed based on the preliminary tests and analyses performed. A complete coating damage was not noticed for the majority of the coatings deposited onto nitride and sialon ceramics, which signifies very goods tribological properties of the coatings deposited. Singular coating damages reaching up to the substrate material were only noticed in case of a Ti(C,N) coating deposited onto the studied substrate (Fig. 10). The material of the counterspecimen used and of the damaged coating is, in rare cases of the examined PVD coatings, adhering, which immediately influences variations in the friction factor values. The counterspecimen material adheres most to a Ti(C,N)+(Ti,Al)N coating deposited onto a nitride ceramics substrate. It was confirmed, after analysing the friction factor attained in the tests undertaken, that the friction factor value varies between 0.2 to 0.6 (Fig. 11).

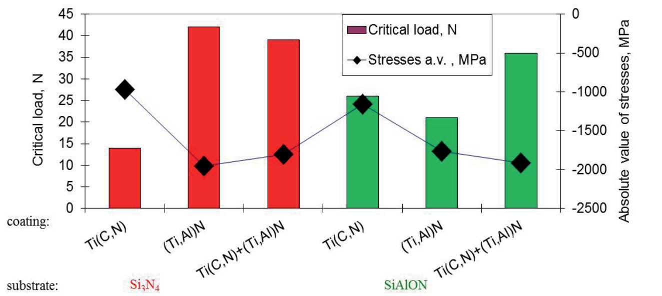


Fig. 9. Comparison of residual stresses and critical load of investigated coatings deposited on different substrates

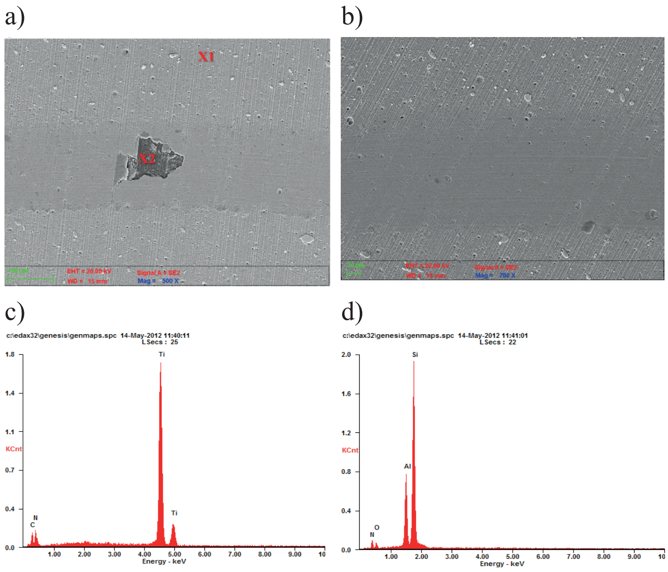


Fig. 10. a),b) Trace of tribological damage on the surface of the Ti(C,N) coating deposited onto a nitride ceramics substrate and diagrams of energy of backscatter X-ray radiation from the microarea: c) X1, d) X2

An antiwear effect of (Ti,Al)N and Ti(C,N)+(Ti,Al)N coatings on the life of indexable inserts was pointed out as a result of grey cast iron rolling tests carried out with Si_3N_4 and SiAlON tool ceramics with the deposited PVD coatings. The highest cutting edge life of T=14 min was achieved for the (Ti,Al)N coating deposited onto a substrate of Si_3N_4. No extended cutting edge life was seen for other indexable inserts with the deposited PVD coatings, because cutting edge destruction takes place at the same time as for an uncoated insert. The poor adhesion of such coatings to the substrate contributes to this fact probably, wear resistance results were satisfactory, though. The tested tools show the wear corresponding to the abrasion and adhesion mechanism during such cutting test (Fig.12).

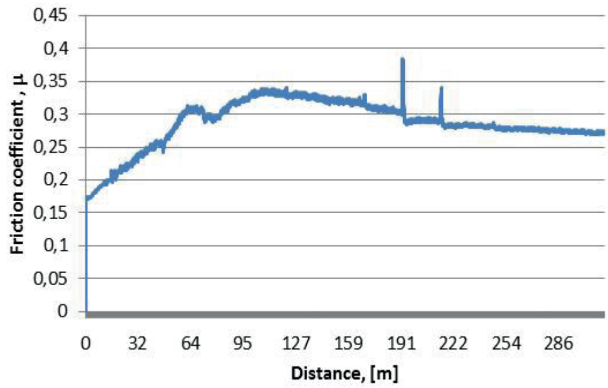


Fig.11. Diagram of friction coefficient according to the friction path during the pin-on-disc test for Ti(C,N) coating deposited onto nitride ceramic

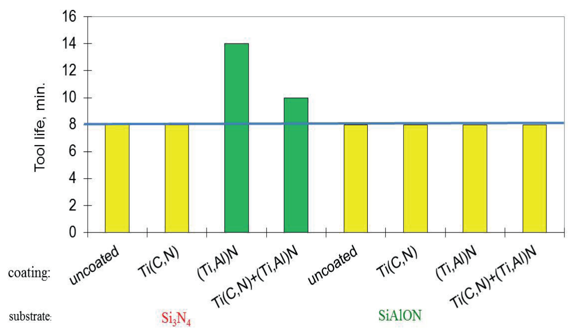


Fig. 12. Values of the life of cutting edges T made of sialon and nitride tool ceramics uncoated and coated with the tested coatings determined in a continuous rolling test of grey cast iron, the cutting speed of v_c = 400 m/min.

5. Summary

Examinations with a scanning electron microscope clearly show a column-like structure of the individual layers deposited into the studied substrates made of tool ceramics.

It can be concluded when evaluating the differences in the texture of the coatings achieved with the PVD method that – most generally – the PVD method supports the creation of texture <111> in the scope of the analysed, manufactured coatings and deposition conditions.

Qualitative correlation between the value of stresses, hardness and adhesion is indicated by the outcomes of the pursued examinations of internal macrostresses for the analysed coatings. The growth of compressive stresses contributes to higher microhardness and better adhesion and wear resistance of the studied coatings.

The low relative values of internal stresses and good adhesion, high hardness and very good wear resistance – noticeable especially for nitride ceramics with (Ti,Al)N and Ti(C,N)+(Ti,Al)N coatings, are extending the useful life of a cutting edge of the studied inserts.

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