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Comparison of nanostructure and duplex PVD coatings deposited onto hot work tool steel substrate

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ABSTRACT

Purpose: The aim of the paper was the investigation of the structure and the mechanical properties of the duplex TiN/(Ti,Al)N coating and the nanostructure TiAlN coating deposited by PVD technology onto hot work tool steel substrate.
Design/methodology/approach: The surfaces' topography and the structure of the PVD coatings were observed on the scanning electron microscopy. Diffraction and thin film structure were tested with the use of the transmission electron microscopy. The microhardness tests were made on the dynamic ultra-microhardness tester. Tests of the coatings' adhesion to the substrate material were made using the scratch test.
Findings: The duplex and nanostructure coatings demonstrate high hardness and very good adhesion. The critical load L_{C2} (coating delamination) lies within the range 80-85 N, depending on the coating type. It was found out that the duplex TiN/(Ti,Al)N coating show the best adhesion to the substrate material.
Practical implications: The very good mechanical properties of the duplex and nanostructure PVD coatings make them suitable in industrial applications.
Originality/value: The investigation results will provide useful information to applying the duplex and nanostructure PVD coatings for the improvement of mechanical properties of the hot work tool steels. The very hard and antiwear PVD coatings deposited onto hot work tool steel substrates are needed.
Keywords: Thin and thick coatings; Nanostructure coatings; Duplex coatings; Mechanical properties

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1. Introduction

The contemporary technologies of materials forming employed in the machining, plastic forming, casting, and also polymer materials forming domains call for using more and more efficient tool materials. The hot work tool steels are the commonly used material. This is due mostly to the complexity of wear processes leading to the tool final failure and often to the tool complex geometry, making it difficult to make it with any other methods than machining [1-6]. Life of the tools made from the hot-work tool steels depends on the heat treatment carried out correctly and the right service conditions, and also on employment of the appropriate cutting-tool lubricants. Extension of the tool life may be also attained by employing the relevant thermo-chemical treatment and development of layers on the tool working surfaces, improving their service properties. Among the surface engineering techniques employed in the last two decades, gas and plasma nitriding, PVD hard coatings deposition, and duplex treatments are the most popular methods reported in the literature [7-10]. Tools made from the hot work tool steels display much better service properties when the nitride coating is developed on them in various nitriding processes, especially in the plasma nitriding process. The plasma nitriding is carried out in the broad temperature range (400-590 ºC), which makes it possible to obtain varying growth of the surface layers, depending on the process conditions, and also on the chemical composition of the substrate. Deposition of hard wear resistant coatings in the PVD processes features the intensively developed research area in the field of improvement of the service properties of tools made from the hot-work tool steels, like employment of the duplex surface treatment - consisting mostly in combining the plasma nitriding and the PVD processes used successfully for the hot work tool steels. The duplex surface treatment reduces the hardness gradient between the coated surface and substrate resulting, thus, in an improved adhesion and an increase of the durability of the tools [11-15].

While selecting the coating material, we encounter a barrier caused by the fact that numerous properties expected from an ideal coating is impossible to be obtained simultaneously. For example, an increase of hardness and strength causes the reduction of the coating's ductility and adherence to the substrate. The application of the nanostructure coatings is seen as the solution of this issue [16, 17]. According to the Hall-Petch equation, the strength properties of the material rise along with the reduction of the grain size. In case of the coatings deposited in the PVD processes, the structures obtained, with grain size ~10 nm cause the obtainment of the maximum mechanical properties. Coatings of such structure present very high hardness >40 GPa, ductility, stability in high temperatures, etc. [18-20].

The aim of this work is the investigation of structure and the mechanical properties of the duplex TiN/(Ti,Al)N coating and the nanostructure TiAlN coating deposited by PVD technology onto hot work tool steel substrate. The investigation results will provide useful information to understanding and applying of these PVD coatings for the improvement of mechanical properties of hot work tool steels.

2. Investigation methodology

The examinations have been made on specimens of hot work tool steel (56 HRC, 30×5 mm) deposited in the PVD process.

Specimens were subjected to heat treatment consisting of quenching and tempering; austenizing was carried out in a vacuum furnace at 1020 °C with a soaking time of 0.5 h. Two isothermal holds were used during heating up to the austenizing temperature, the first at a temperature of 640 °C and the second at 840 °C. The specimens were tempered twice after quenching, each time for 2 hours at the temperature of 560 °C and next at 510 °C. To ensure proper quality, the surfaces of the steel specimens have been subjected to mechanical grinding and polishing (R_a=0.03 μm). After the heat treatment the TiAlN PVD nanostructure coating was deposited. After the heat treatment one of the samples were nitrided, the following plasma nitriding (PN) conditions were applied: gas composition - 90%N_2+10%H_2 surface temperature – 550 ˚C, treatment time – 3 h, after nitiriding the samples were polished to a roughness R_a = 0.08 μm, than the TiN/(Ti,Al)N PVD coatings were deposited.

The investigations of diffraction and structure of thin foils were made with a JEOL JEM 3010UHR transmission electron microscope at an accelerating voltage of 300 kV. Thin foils were made by mechanical grinding and further ion polished using the Gatan apparatus. Observations of surface and structures of the deposited coatings were carried out on cross sections in the scanning electron microscope SUPRA 25 and ZEISS (SEM). Detection of secondary electron was used for generation of fracture images, the accelerating voltage was 15 kV, and the maximum magnification was 30 000×. The phase composition of the coatings were determined on the DRON-2.0 X-ray diffractometer, using the filtered radiation of the Co anode lamp, powered with 40 kV voltages at 20 mA heater current. The distribution of the concentrations of the elements along the thickness of the coating was determined using the GDOS method – glow discharge optical emission spectrometry, employing a SDP 750A spectrometer made by LECO. The sputtering parameters were: cathode voltage 700 V, ion current 25 mA.

The evaluation of the adhesion of coatings to the substrate was made using the scratch test with the linearly increasing load, the test were made by the CSEM REVETEST scratch tester. The critical forces at which coating failures appear, called the critical load L_c, were determined basing on the acoustic emission AE registered during the test and microscope observations. Observation of the damage developed in the scratch test on a scanning electron microscope Opton DSM 940. During the adhesion scratch tests of the coatings one has observed damages which P. Burnett divided as follows [12, 13]:

- spalling failure,
- buckling failure,
- chipping failure,
- conformal cracking,
- tensile cracking.

Hardness tests of the investigated PVD coatings were made using Vickers micro-hardness testing method. The thickness of coatings was determined using the "kalotest" method, measuring the characteristic of the spherical cap crater developed on the surface of the coated specimen tested.

3. Discussion of results

The morphology of the investigated nanostructure and duplex PVD coatings deposited onto hot work tool steel is characterised by a significant inhomogeneity connected with the occurrence of multiple drop-shaped micro-particles on their surface and also with pits developed by falling out by some of these drops (Figs. 1, 2). Metallographic examinations of coatings fractures show that TiAlN and TiN/(Ti,Al)N coatings have compacted, columnar structure and the TiN/(Ti,Al)N duplex coatings show laminar structure (Figs. 3, 4). The fractographic test of the TiAlN coating indicate a sharp interface zone between the substrate and the coatings. It has been found out that the investigated PVD coatings deposited onto hot work tool steel are characterised by a uniform thickness.

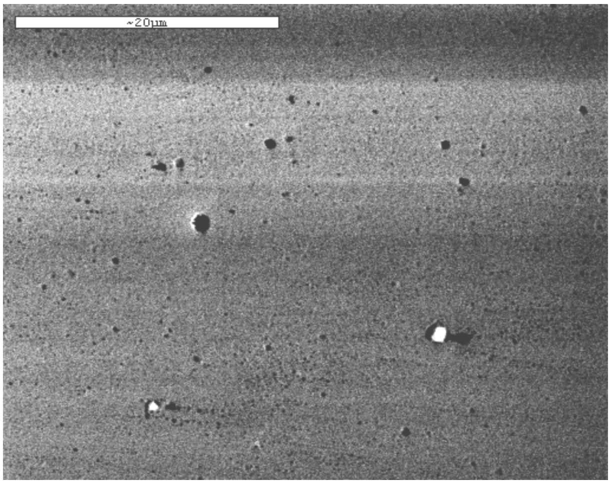


Fig. 1. Topography of the TiN/(Ti,Al)N PVD coating deposited onto plasma nitrided hot work tool steel

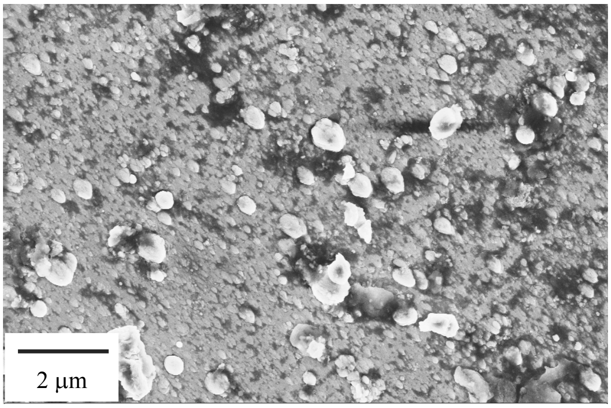


Fig. 2. Topography of the TiAlN PVD nanostructure coating deposited onto hot work tool steel

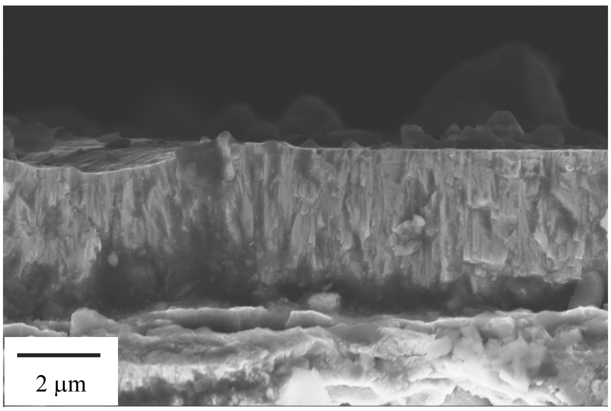


Fig. 3. Fracture of the TiN/(Ti,Al)N PVD coating deposited onto plasma nitrided hot work tool steel

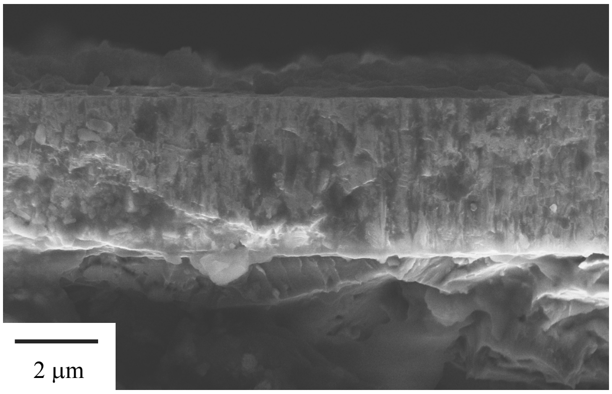


Fig. 4. Fracture of the TiAlN PVD nanostructure coating deposited onto hot work tool steel

TEM investigations of the TiAlN nanostructure and TiN/(TiAl)N duplex coatings (Figs. 5, 6) shows fine crystallites with an average size is about 15-50 nm. Generally, there are no premises that might suggest the epitaxial growth of at least fragments of the examined coatings.

X-ray diffraction patterns of the TiAlN PVD nanostructure coating deposited onto hot work tool steel (Fig. 7) shown that the coatings contain only one fcc phase. In case of the TiAlN phase the diffraction lines are shifted to the larger deflection angles. On the X-ray diffraction patterns for the TiN/(Ti,Al)N PVD coatings deposited onto plasma nitrided hot work tool steel, the appearance of the reflexes coming from substrate material tempered martensite, has been ascertained (Fig. 8). It develops from a little thickness of the deposited coating, smaller than the X-rays penetration depth into the material. The TiAlN and TiN/(Ti,Al)N coatings show a privileged crystallographic orientation. In case of the TiN/(Ti,Al)N duplex coating (Fig. 8), the identified reflexes come from the nitride coating Fe_3N and Fe_4N.

The changes of the chemical concentration of the coating constituents and substrate material upon tests carried out on the glow-discharge optical emission spectroscope are present on the Figures 9, 10.

Investigations made using the GDOS indicate also to the existence of the transition zone between substrate material and coating, improving adhesion of the deposited coatings to the substrate. In the transition zone the concentration of elements included in the substrate grows and the concentration of elements constituting the coatings decreases rapidly. Its development may be also connected with the effect of the high energy ions causing transferring the elements in the joint zone, increase of desorption of the substrate surface, and development of defects in the substrate.

One should stress that the investigation results obtained with the GDOS cannot be unequivocally interpreted due to the inhomogeneous vaporization of the specimen material during the examination.

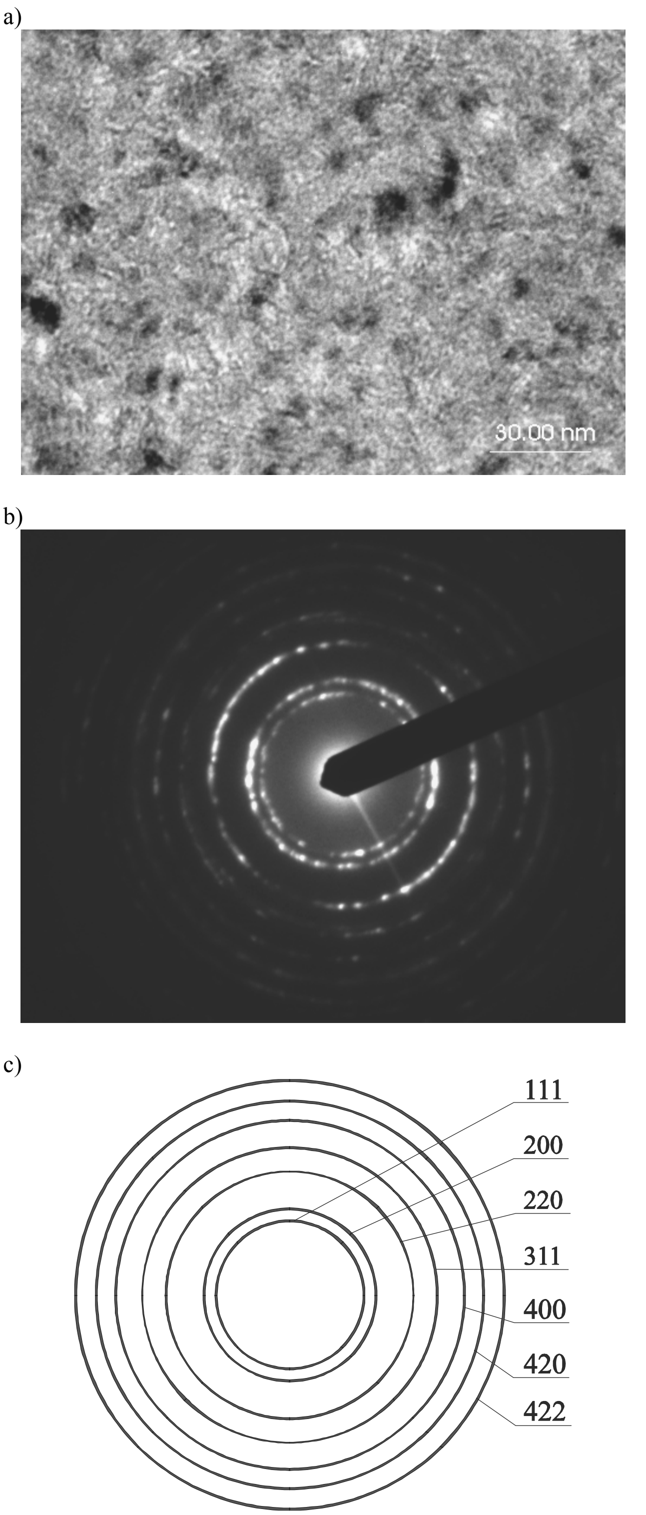


Fig. 5. a) Structure of the thin foil from the TiAlN coating deposited onto the hot work tool steel, b) diffraction pattern from the area as in figure a, c) solution of the diffraction pattern indicating (Ti,Al)N phase

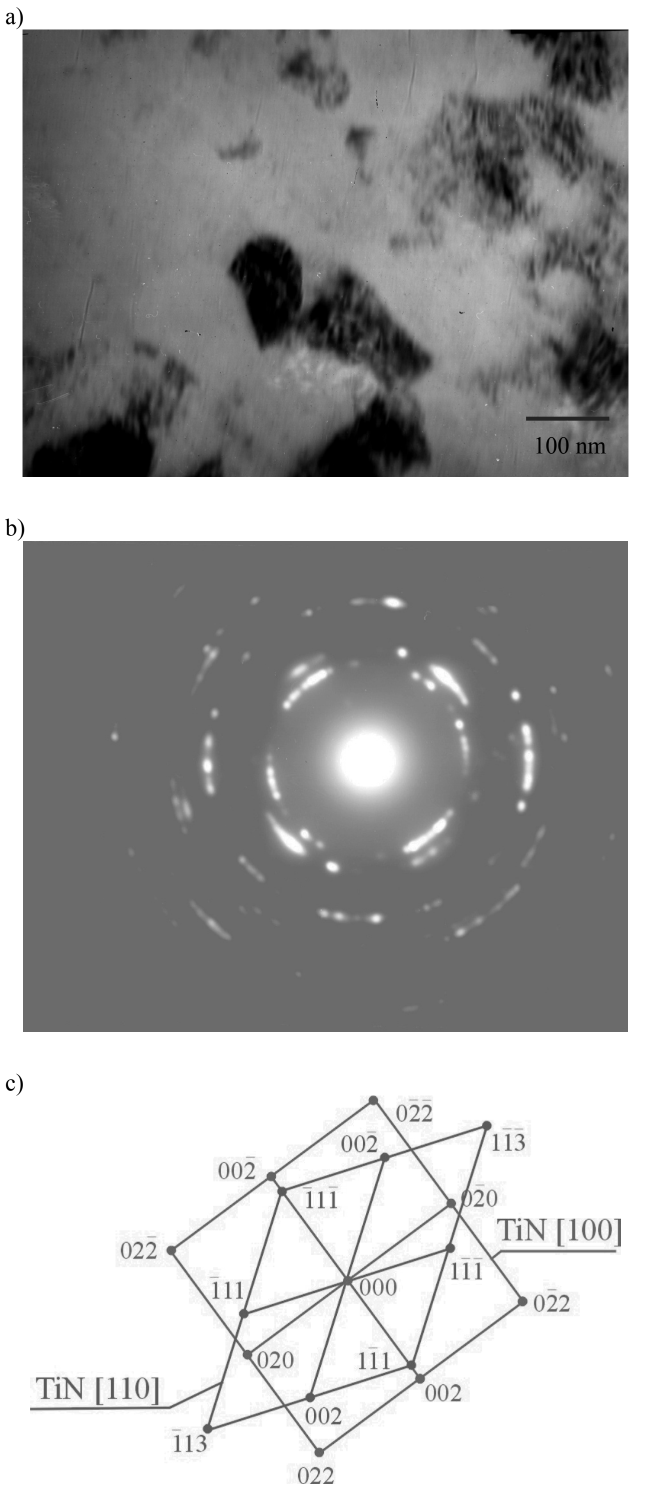


Fig. 6. a) Structure of the thin foil from the TiN/(Ti,Al)N coating deposited onto plasma nitrited hot work tool steel, b) diffraction pattern from the area as in figure a, c) solution of the diffraction pattern indicating TiN phase

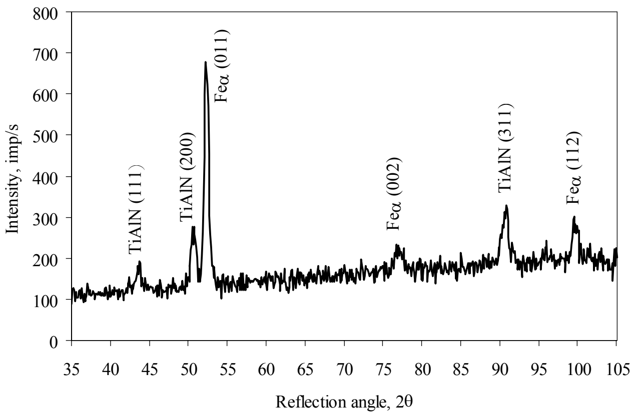


Fig. 7. X-ray diffraction pattern of the TiAlN nanostructure coating deposited onto the hot work tool steel

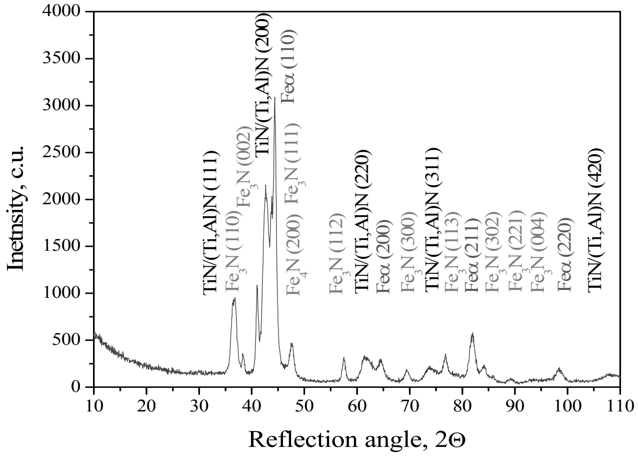


Fig. 8. X-ray diffraction pattern of the TiN/(Ti,Al)N PVD coatings deposited onto plasma nitrided hot work tool steel

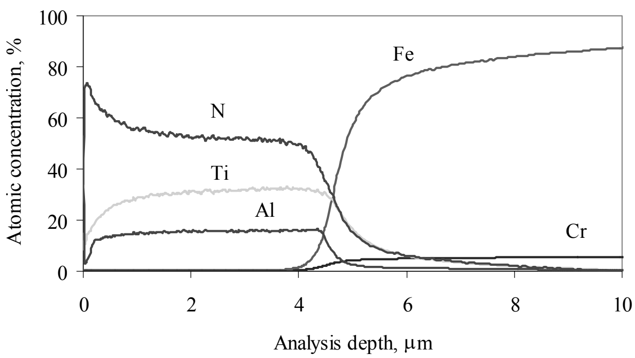


Fig. 9. Changes of concentrations of constituents of the TiAlN PVD nanostructure coating and of the substrate materials

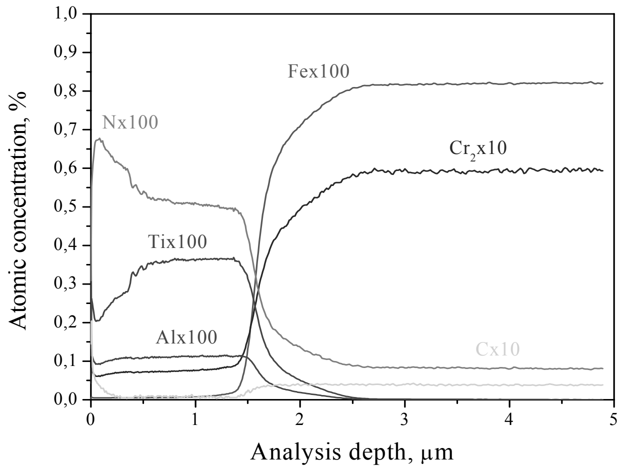


Fig. 10. Changes of concentrations of constituents of the TiN/(Ti,Al)N PVD coatings and of the substrate materials

The critical load values L_C, that are characterized by the adhesion of the investigated PVD coatings to the substrate from the hot work steel are presented in Table 1. The critical loads L_{C1} and L_{C2} were determined by scratch testing (Figs. 11-14).

Table 1.
Critical loads for TiAlN nanostructure coating deposited onto heat treated hot work tool steel and for TiN/(Ti,Al)N coating deposited onto plasma nitrited hot work tool steel

Coating type	Critical load [N]	
	L_{C1}	L_{C2}
TiAlN	35	80
TiN/(Ti,Al)N + PN	56	85

The first critical load L_{C1} corresponds to the point at which first damage is observed; the first appearance of microcraking, surface flaking outside or inside the track without any exposure of the substrate material – the first cohesion-related failure event. This first damage has the shape of an interfacial shell-shaped spallation. L_{C1} corresponds to the first small jump on the acoustic emission signal, as well as on the friction force curve. The second critical load L_{C2} is the point at which complete delamination of the coating starts; the first appearance of cracking chipping, spallation and delamination outside or inside the track with the exposure of the substrate material – the first adhesion-related failure event. After this point, all the acoustic emission, and friction force signals become noisier. It has been found out, on the basis of on the determined Lc (AE) values and on the developed failures metallographic examinations that multilayer TiN/(Ti,Al)N coatings have very good adhesion to the substrate from the nitrided hot work tool steels, whereas the TiAlN nanostructure coatings adhesion reaches the lowest value. The difference consists in the location of these spalling defects. In case of the TiN/(Ti,Al)N coating the spalling defects begin at the load value of about 56 N. Next, cracks and coating stretches, develop on the scratch bottom, and finally the total coating delamination on the scratch bottom takes place – 85 N.

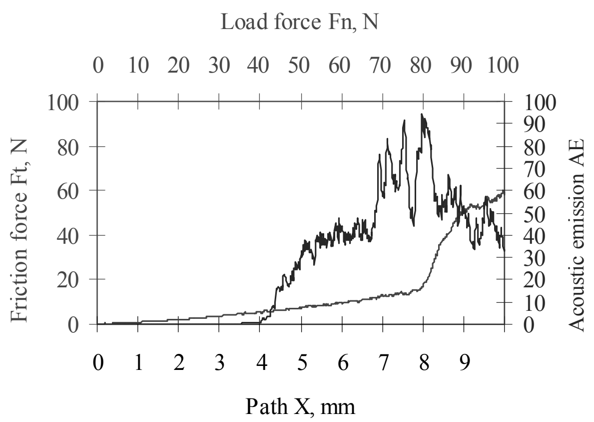
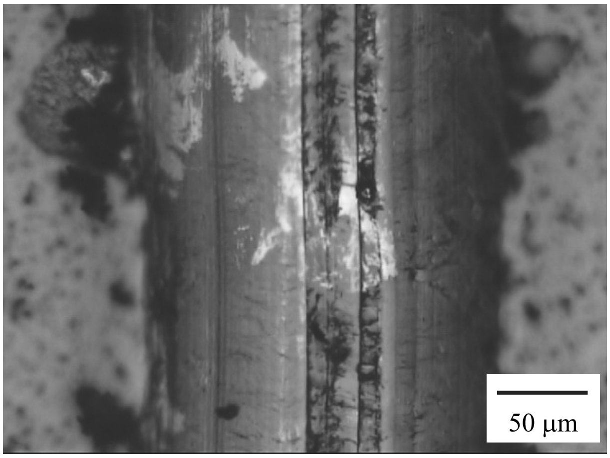


Fig. 11. Diagram of the dependence of the acoustic emission (AE) and friction force F_t on the load for the nanostructure TiAlN coating

a)



b)

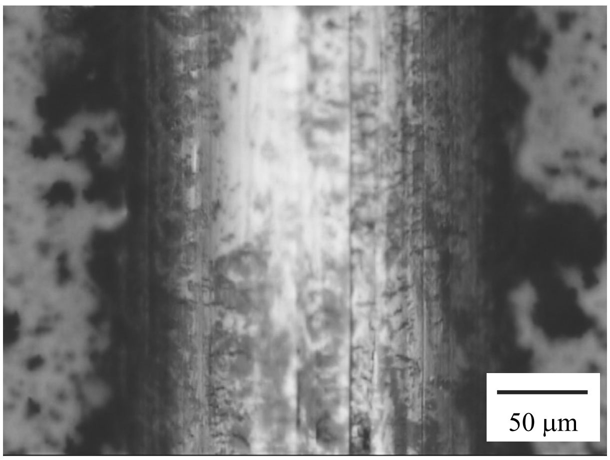
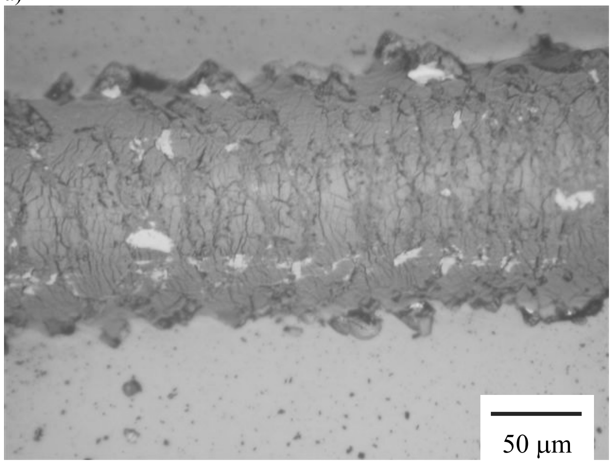


Fig. 12. Scratch failure pictures of the nanostructure TiAlN coating on hot work tool steel substrate at: a) L_{C1}, b) L_{C2}

a)



b)

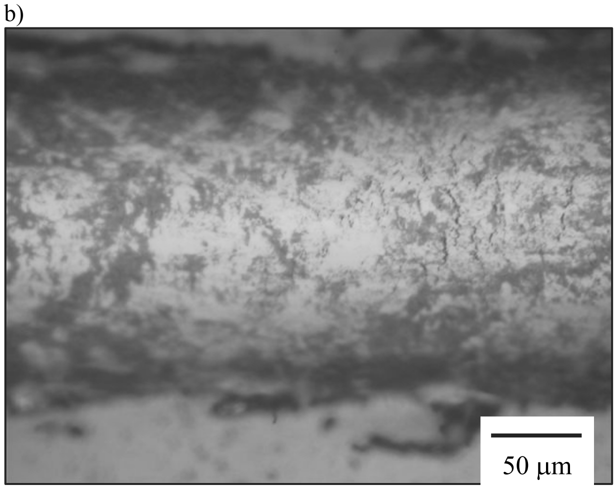


Fig. 13. Scratch failure pictures of the TiN/(Ti,Al)N coating on plasma nitrided hot work tool steel substrate at: (a) L_{C1}, (b) L_{C2}

The employment of the EDS analyser on the scanning microscope has let it to reveal, that in case of the TiN/(Ti,Al)N coating delamination occurs from the initially deposited titanium sublayer. The analysis of the test results makes it possible to state that in case of the nanostructure TiAlN coating the numerous spalling defects of the scratch edges begin at load of 35 N and than spalling defect at the edge gets deeper and next coating delamination occurs – L_{c2}-80 N. The TiN/(Ti,Al)N PVD coating on the plasma nitrided hot work tool steel is characteristic of a better adhesion to the substrate material, compared to the adhesion of the TiAlN nanostructure coating on the heat treated steel, this is caused not only by adhesion but also by the thicker interface between the coating and the substrate and by the 148 µm thick nitrided layer with 1480 $HV_{0.1}$ hardness, featuring the PVD coating substrate.

a) b) c)

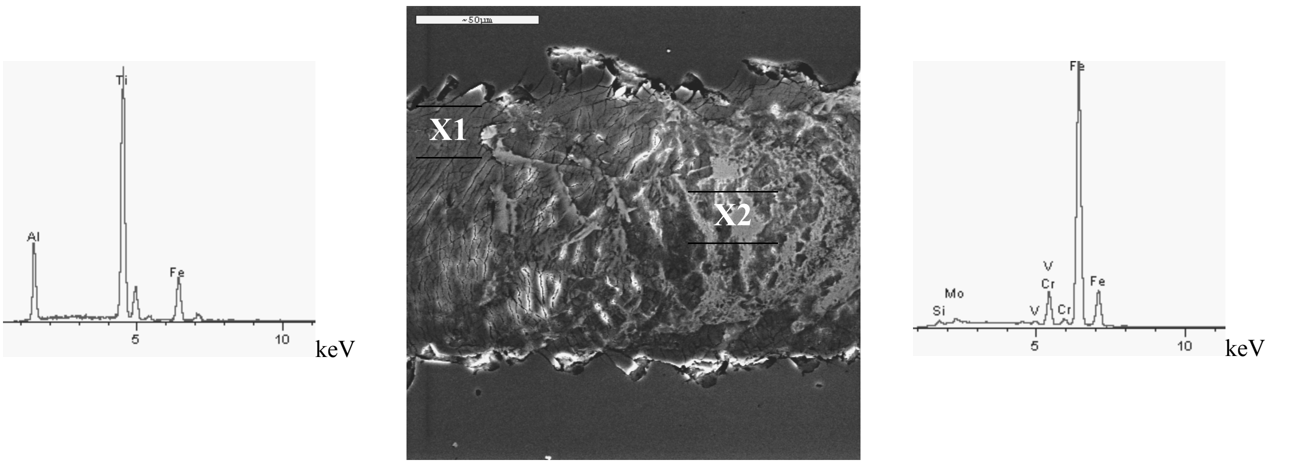


Fig. 14. Scratches with critical load: a) X1 from areas with coating, b) L_{C2} – partial delamination of TiN/(Ti,Al)N coating; EDS spectra, c) X2 from areas without coating

4. Conclusions

Basing on the investigation results the following conclusion were arrived at:

- the compact structure of the coatings without any visible delamination was revealed on the scanning electron microscope. The investigated TiAlN and TiN/(Ti,Al)N coatings show columnar structure;
- the TiN/(Ti,Al)N PVD coating on the plasma nitrided hot work tool steel is characteristic of a better adhesion (L_{C2}=85 N) to the substrate material, compared to the adhesion of the nanostructure TiAlN coating on the heat treated steel (L_{C2}=80 N), this is caused not only by adhesion but also by the thicker interface between the coating and the substrate and by the 148 µm thick nitrided layer with 1480 $HV_{0.1}$ hardness, featuring the PVD coating substrate;
- improved mechanical properties of the substrate in the plasma nitrided layer contribute to the coatings fragmentation reduction due to plastic deformation, their conformal cracking, spalling, chipping and delamination, contributing to improvement of the coating adhesion parameters and wear resistance;
- the investigations made using the glow discharge optical emission spectrometer GDOS indicate to the existence of the interface between the substrate material and the coating resulting in improvement of the adhesion of the coatings deposited to the substrate.

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