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Comparison of the CrN and TiN/(Ti,Al)N PVD coatings deposited onto plasma nitrited steel

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ABSTRACT

Purpose: The paper presents the results of the mechanical and tribological properties of the CrN and TiN/(Ti,Al)N PVD coatings deposited onto plasma nitrided hot work tool steel X37CrMoV5-1 type.

Design/methodology/approach: Tests of the coatings' adhesion to the substrate material were made using the scratch test. The surfaces' topography, structure of the PVD coatings were observed on the scanning electron microscopy. Wear resistance tests with the pin-on-disc method were carried out on the CSEM THT (High Temperature Tribometer).

Findings: The results showed that the duplex TiN/(Ti,Al)N coating exhibited higher hardness, very good adhesion and better wear resistance in the elevated temperature as compared to the duplex CrN coating.

Practical implications: The investigation results will provide useful information to applying the duplex PVD coatings for the improvement of mechanical properties of the hot work tool steels.

Originality/value: The very hard and antiwear PVD coatings deposited onto hot work tool steel substrate are needed.

Keywords: Thin and thick coatings; PVD coatings, Duplex coatings; Mechanical properties

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

Metalworking industry has shown interest in improving tools used in hot working process: metal die casting, hot extrusion, hot forging and rolling. The performance of many products and their components depends not only on the possibility of transferring mechanical loads throughout the section of the element with the material or its physical and chemical properties, but very often on the structure and properties of surface layers. It is also because of economic considerations that the layer processing technology is to be applied to ensure the required functional properties while using as cheap as possible materials for the core element, this element being required to generally have a lower performance. A wide selection of currently available types of coatings and their deposition technology is the result of an increasing demand for modern methods of surface modification and protection for tools, parts of machine and other items [1-8].

One of the most frequently applied method of tool life improvement is PVD technique. Thin hard PVD coatings are today employed in vast number of applications for reducing friction and wear of tools and mechanical components. PVD coatings have also been used for selected hot-working processes [9-13].

The CrN coating prepared through physical vapour deposition is often used to prolong the service life of workpieces due to its high adhesion strength with susbstrate, good hardness and thermal conductivity. The TiN(Ti,Al)N coating can also provide wear and oxidation resistance, especially at high temperature [14-18] .

However, application of PVD hard coatings to the relatively soft substrate cannot guarantee the optimal tribological performance. The best results in protection of tools made of hot work steel were obtained with duplex treatment procedure. Duplex treatment is combined thermo-chemical treatment of the tool followed by PVD hard coatings deposition. Surface layers obtained in this way display properties characteristic of both types of treatment, ensuring simultaneously the quasi-gradient changes of structure and properties of the surface layers of the hot-work tools. The thermo-chemical treatment like plasma nitriding provides better mechanical support to the hard and mainly brittle coating. Duplex treatment has proven successful in improving wear, fatigue and corrosion resistance and the load carrying capability of hot work steel substrates [19-22].

The paper presents the results of the project focused on the investigation of the structure, mechanical and tribological properties of CrN and TiN/(Ti,Al)N PVD coatings deposited onto plasma nitrided hot work steel X37CrMoV5-1 type.

2. Investigation methodology

The PVD coatings were deposited onto X37CrMoV5-1 type hot work steel substrate. The samples were quenched at 1020°C and tempered at 550°C to hardness 55 HRC. After the thermal treatment, the samples were grounded and polished, another these samples were nitrided, the following plasma nitriding conditions were applied:

- gas composition - 90%N_2+10%$H_{2,}$
- surface temperature - 550°C,
- treatment time - 3 h.

After nitiriding the PVD coatings were deposited. The CrN coatings were prepared in BALZERS BAI 730 deposition system by ion plating PVD process at 450°C temperature, while TiN/(Ti,Al)N multilayer coating was deposited by magnetron sputtering in CemeCon apparatus at the temperature 450°C.

The surface roughness of the polished specimens and roughness of the PVD coatings were measured on the Taylor-Hobson Form Talysurf Series 2 profilometer. The parameter R_a was assumed as a quantity describing the surface roughness. Hardness test of the investigated specimens from hot work steel in the heat treated state has been made using Rockwell method. The distribution of microhardness in the nitriding layer was measured using Vickers micro-hardness testing method using a load of 0.981 N. Hardness tests of the investigated PVD coatings were made using Vickers micro-hardness testing method with a load of 10mN which ensures the limited indenter penetration depth to eliminate the substrate influence. The tests were carried out on the Fischerscope micro - hardness tester. The examinations of the microstructure of the nitrided layer were made in the scanning electron microscope (DSM 940 Opton) on the etched cross-sections. The surfaces' topography of the investigated PVD coatings was observed on the scanning electron microscope (SEM) Opton DSM 940. The specimens with the notch cut were cooled in liquid nitrogen before breaking in order to observe their structure on transverse fractures on the Opton DSM 940 SEM. The phase composition of the plasma nitrided hot work steel and coatings was determined using the Dron 2.0 diffractometer, using the X-ray radiation with the Co anode. The measurements were made in the 2Θ angle ranging from 10 to 110°. 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 750 A 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 crytical forces at which coating failures appear, called the critical load L_c, was determined basing on the acoustic emission AE registered during the test and microscope observations for five critical forces: L_{c3} - flaking on the scratch edge, L_{c4} - coating partial delamination, L_{c5} - coating total delamination and $L_c(F_t)$ - sudden increase of the scratching force. The character of the defects was determined basing on observation performed on the scanning electron microscope Opton DSM 940. Wear resistance tests with the pin-on-disc method were carried out on the CSEM THT (High Temperature Tribometer) device at the room temperature and at the temperature of 500°C. The Al_2O_3 - corundum ball of the 6 mm diameter was used as counter-specimen. During the pin-on-disc test carried out at the room temperature and at 500°C the stationary ball was pressed with the load of 7.0 N to the disc rotating in a horizontal plane.

The rotational speed of the disc with the specimen was 50 cm/s. The friction coefficient between the ball and disc was measured during the test. The friction radius and number of rotation were changed like:

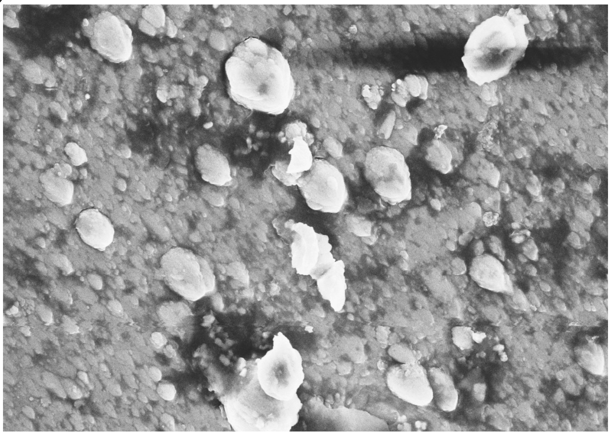
- 1000 revolutions - 20°C - friction radius -10mm
- 7500 revolutions - 20°C - friction radius - 13mm
- 1000 revolutions - 500°C - friction radius - 16mm
- 7500 revolutions - 500°C - friction radius - 17.5mm

Examinations of wear traces developed during the pin-on-disc test were made on the LEICA MEF4A light microscope at 100x magnification. Wear traces profiles were measured on the Taylor - Hobson Form Talysurf 120L laser profilometer in eight directions (every 45°).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 maximal hardness of the 3h long plasma nitride coating onto the hot work tool steel X37CrMoV5-1 is 1478 $HV_{0,1}$ with the increase of the distance from the surface, microhardness of the investigated nitride coating goes down to 612 $HV_{0,1}$ specific for the core. It has been stated, on the basis of the observation of microstructure of the nitride coating on the scanning microscope, that it is characterized by a homogenous, compact and zonal structure. The results of research done with the method of X-ray phase analysis confirm the occurrence of nitride phases in the structure of surface coating, obtained through plasma nitriding.

a)



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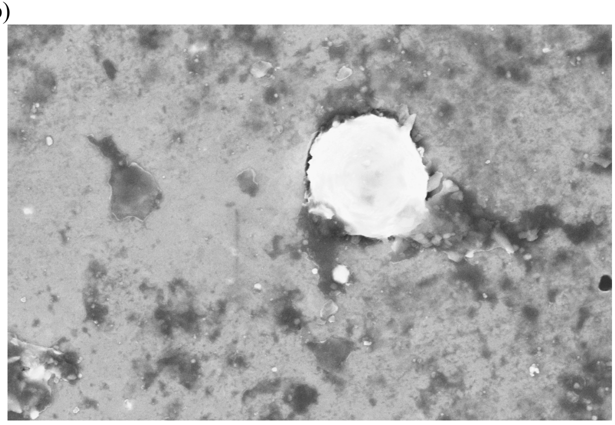
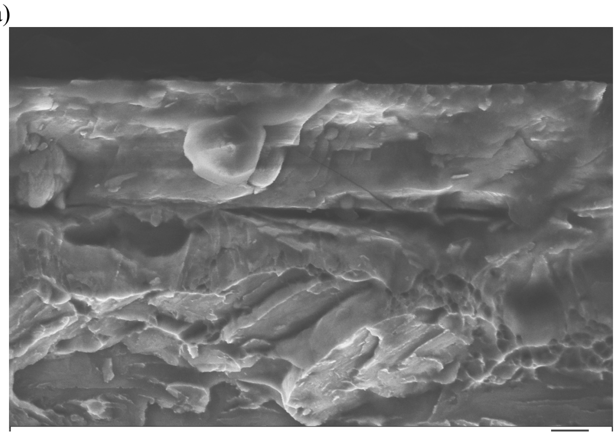


Fig. 1. Topography of the PVD coatings deposited onto plasma nitrided hot work steel X37CrMoV5-1 type: a) CrN coating, b) TiN/(Ti,Al)N coating

As a result of the metallographic examinations made on the SEM it has been found out that the morphology of the investigated PVD coatings deposited onto plasma nitrided hot work steel X37CrMoV5-1 type 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. The presence of these defects was observed in the largest scale in case of CrN monolayer coatings when the presence in TiN/TiAlN multilayer coating was the smallest one. Most probably the limited presence of defects in case of multilayer TiN/TiAlN coating results from their deposition process conditions. Periodically changing of metallic vapor sources in the PVD process of coatings deposition do not allow to build up micro droplets deposited in the early stage of deposition of each layer as well as hide the pits after their falling out (Fig. 1).

The results of this investigation correspond with the results of roughness and value of the friction coeffiction. Metallographic examinations of coatings fractures show that TiN/(Ti,Al)N coatings have compacted, columnar structure while the CrN coating has a compacted submicrocrystalline structure. Examinations of the fracture surface of the TiN/(Ti,Al)N coatings indicate their laminar structure. It has been found out that the investigated PVD coatings deposited onto plasma nitrided hot work steel X37CrMoV5-1 type are characterized by a uniform thickness (Fig. 2).

a)



b)

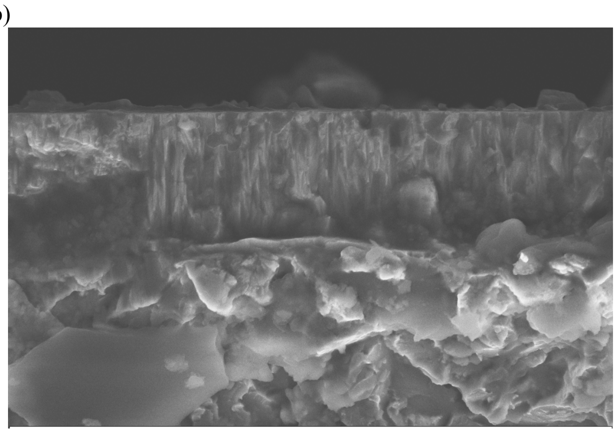


Fig. 2. Fracture of the investigated PVD coatings deposited onto plasma nitrided hot work steel X37CrMoV5-1 type: a) CrN coating, b) TiN/(Ti,Al)N coating

The X-ray phase analysis confirms the occurrence of nitride coating and the CrN and TiN/(Ti,Al)N coatings onto the examined hot work tool steel. On the X-ray diffraction patterns for all the investigated coatings as well as the nitride coating, the appearance of the reflexes coming from substrate material martensite, has been ascertained. It develops from a little thickness of the deposited coating, smaller than the X-rays penetration depth into the material. The monolayer CrN coating and TiN/(Ti,Al)N multilayers show a privileged crystallographic orientation. In case of PVD coated steels the identified reflexes come from the nitride coating Fe_3N and Fe_4N (Fig. 3).

a)

b)

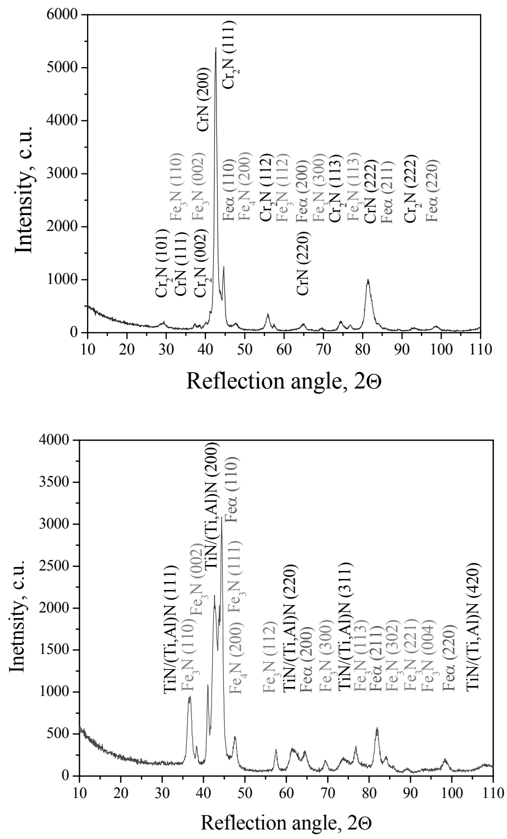


Fig. 3. The X-ray diffraction pattern of the PVD coatings deposited onto plasma nitrited hot work steel X37CrMoV5-1 type: a) CrN, b) TiN/(Ti,Al)N coatings

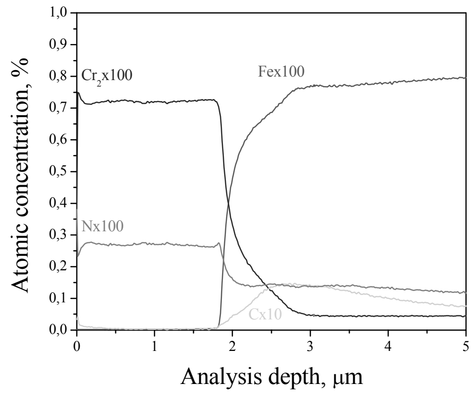


Fig. 4. Changes of concentrations of constituents of the CrN PVD coatings and of the substrate materials

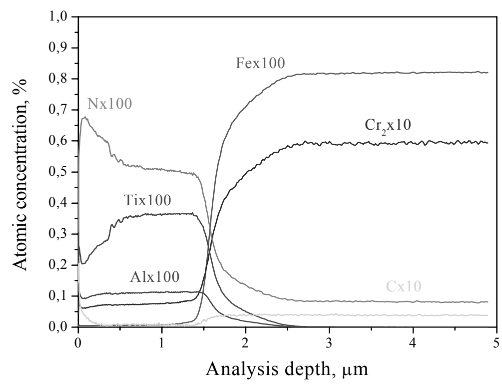


Fig. 5. Changes of concentrations of constituents of the TiN/(Ti,Al)N PVD coatings and of the substrate materials

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 (Figs. 4, 5).

The roughness of the investigated PVD coatings ranges from 0.101 to 0.319 μm. The results of these measurements correspond with the metallographic examinations made on the SEM. The topography of the coatings influences the roughness, which is characterized by heterogeneity in the forms of cavities and elementary particles as well as a little smoothness of the surfaces of the investigated PVD coatings. The microhardness of the PVD coatings was made using a load 10mN, making it possible to eliminate, to the greatest extent, the influence of the substrate on the obtained results. The microhardness ranges from 2443 to $2927HV_{0,001}$. The results of this investigation can be indicated the correlation between hardness and adhesion of the PVD coatings to the substrate material.

It has been found out, on the basis of on the determined L_c(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 tools steels, whereas the CrN coatings adhesion reaches the lowest value (Table 1).

Table 1.
Critical loads for investigated coatings

Coating type	Type of defect/Force [N]				
	L_c (AE)	L_{c3}	L_{c4}	L_{c5}	L_c (F_t)
CrN	37	51	70	86	83
TiN/(Ti,Al)N	56	62	85	110	100

The damage of the coatings commences in all cases with the widespread coating spallation on both edges of the originating scratch (Figs. 6, 7). 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 62 N (Fig. 9). Next, cracks and coating stretches, develop on the scratch bottom, and finally the total coating delamination on the scratch bottom takes place (Fig. 11).

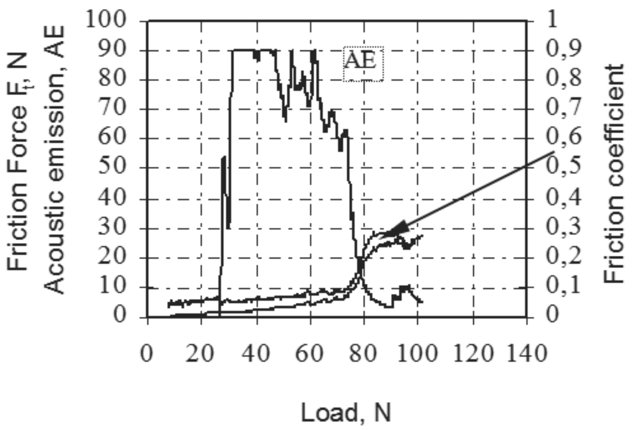


Fig. 6. Diagram of the dependence of the acoustic emission, friction force and friction coefficient on the load for the CrN coating

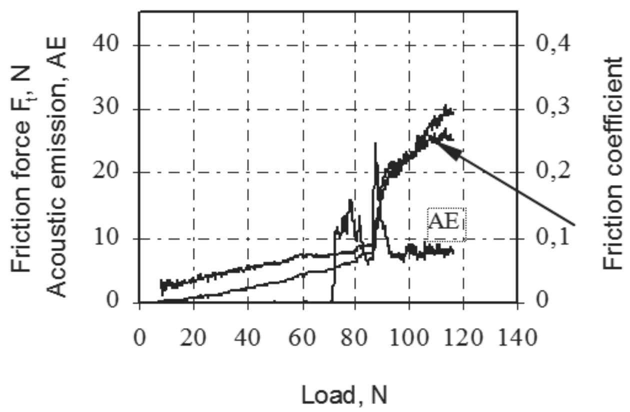


Fig. 7. Diagram of the dependence of the acoustic emission, friction force and friction coefficient on the load for the TiN/(Ti,Al)N coating

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 single-layer CrN coating the numerous spalling defects of the scratch edges begin at load of 51 N (Fig. 8). Spalling defect at the edge gets deeper and next coating delamination occurs (Fig. 10). A weak adhesion of the CrN coating to the substrate may result from its relatively low hardness. As a result of the pressure of the indenter, the plastic deformation of the coating may take place as the softer and more elastic CrN coating undergoes a bigger deformation than the hard, multilayer TiN/(Ti,Al)N coating.

Test results of the investigated PVD coatings adhesion to the substrate from the nitrided hot work tool steel correspond with the results of the wear test.

The investigated coatings were subjected to the pin-on-disc tribological test carried out at room temperature (20°C) and at the temperature elevated to 500°C to determine their wear resistance. Changes of the friction coefficient values between the corundum ball and the examined test piece were recorded during the tests at room temperature and at the temperature of 500°C. It has been stated that the friction coefficient of the plasma nitride steel is about 0.5 for 1000 revolutions. The wear has an adhesive character at the temperature of 20°C for 1000 revolutions. At the temperature of 500°C the friction coefficient grows at the beginning, and on the surface there are craters arisen after the removed coating as a result of friction of the ball against the sample surface.

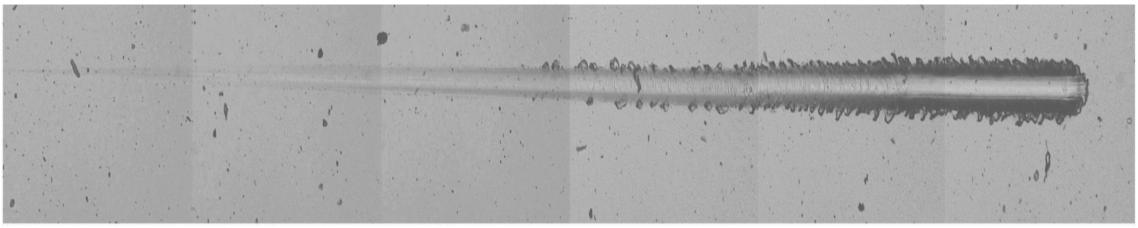


Fig. 8. Scratches with typical delamination of the CrN coating deposited onto plasma nitrited hot work tool steel X37CrMoV5-1 type

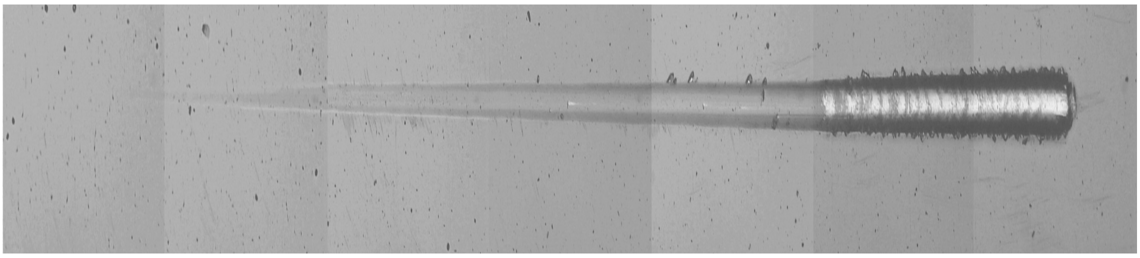
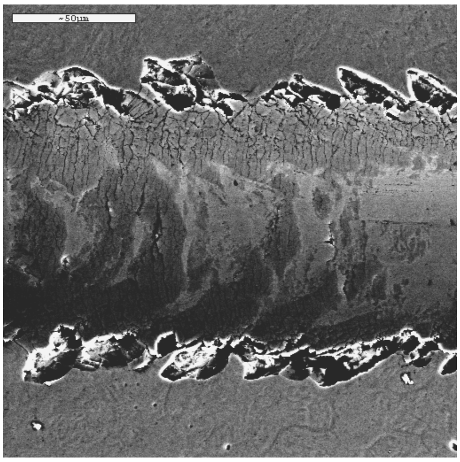
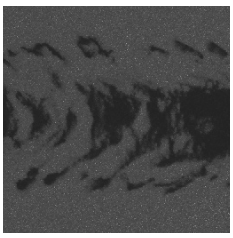


Fig. 9. Scratches with typical delamination of the TiN/(Ti,Al)N coating deposited onto plasma nitrited hot work tool steel X37CrMoV5-1 type

a)



b) Cr



c) Fe

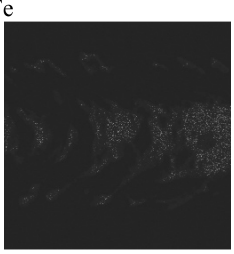


Fig. 10. Scratches with critical load of CrN coating: a) partial delamination L_{c4}, Elemental maps: b) Cr-from areas with coating, c) Fe-from areas without coting

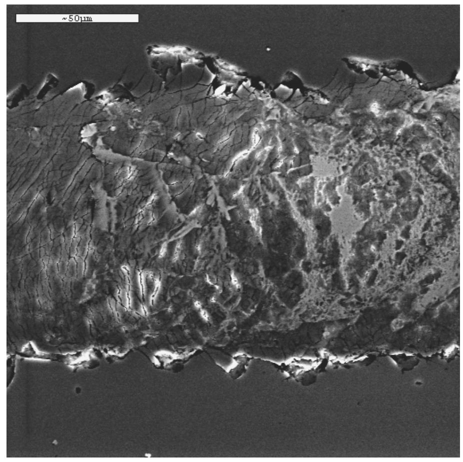
The measured profiles' data were collected and the average profiles of the scratch trace for each of the examined coatings were determined. The width and depth of the wear were measured for the average profile determined in this way. Moreover, the widths of the wear traces developed during the pin-on-disc test on the examined coatings were measured on the scanning electron microscope. At the known wear trace width, the average volume of the material removed due to friction of the corundum ball against the test piece surface can be calculated according to the formula (1):

$$V = \pi * R * D^3 / 6 * r \ [mm^3] \qquad (1)$$

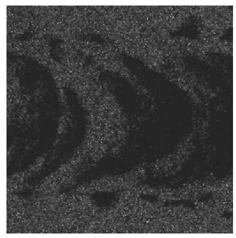
where: V - average volume of the material worn out due to friction, R - friction radius [mm], D - wear trace width [mm], r - ball radius [mm]

It has been noted, on the basis of research done, that the plasma nitrided at 20°C and 500°C temperatures steel underwent the biggest wear. The deposition of the coating significantly increases the wear resistance. One can state, basing on the completed wear measurement results of the PVD coatings on the X37CrMoV5-1 nitrided hot work steel (Table 2), that during the tests at the temperature of 20°C for both 1000 and 7500 revolutions the highest wear resistance was characteristic of the TiN/(Ti,Al)N coating.

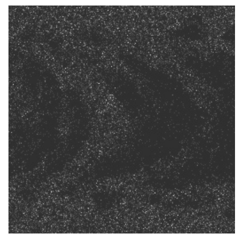
a)



b) Ti



c) Al



d) Fe

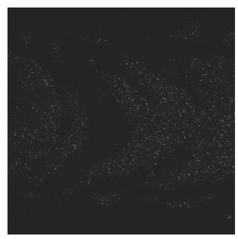


Fig. 11. Scratches with critical load of TiN/(Ti,Al)N coating: a) partial delamination L_{c4}, Elemental maps: b) Ti-from areas with coating, c) Al-from areas with coating, d) Fe-from areas without coating

The TiN/(Ti,Al)N coating proved to perform well in the test conditions for 1000 and 7500 revolutions. Therefore, one can state that both at the room and elevated temperatures the multilayer TiN/(Ti,Al)N coating is characteristic of the best wear resistance, with the single layer CrN coating taking the second place. In case of the TiN/(Ti,Al)N coating deposited onto the X37CrMoV5-1 nitrided hot work steel the very good adherence was revealed to the substrate material compared to the CrN coating. On the basis of the research done one can state that the temperature has been a decisive coefficient in the carried out test. There is the biggest material wear at the temperature of 500°C.

The deposition of PVD coatings onto the nitride tool steel, however, considerably improves its anti-wear properties. The nitride coating improves the adhesion of the examined coatings and consequently their anti-wear properties through the decreasing of the friction coefficient. The evaluation results of the volume of material removed during the pin-on-disc test correspond with the wear trace width measurements made by observations carried out on the scanning electron microscope. The wear trace width values measured on the scanning electron microscope grow with the test temperature, regardless of the number of revolutions made by the test piece (Fig. 12).

Table 2.
Comparison of volume of materials removed during tribological wear for 1000 and 7500 revolutions

Substrate material/Coating type	Volume of materials removed V [mm^3]	
	20°C	500°C
1000 revolutions		
Plasma nitrided hot work steel X37CrMoV5-1 type	0.26375	0.41216
CrN	0.24681	0.40848
TiN/(Ti,Al)N	0.14358	0.30250
7500 revolutions		
Plasma nitrided hot work steel X37CrMoV5-1 type	0.31025	0.69655
CrN	0.30677	0.62388
TiN/(Ti,Al)N	0.22303	0.47413

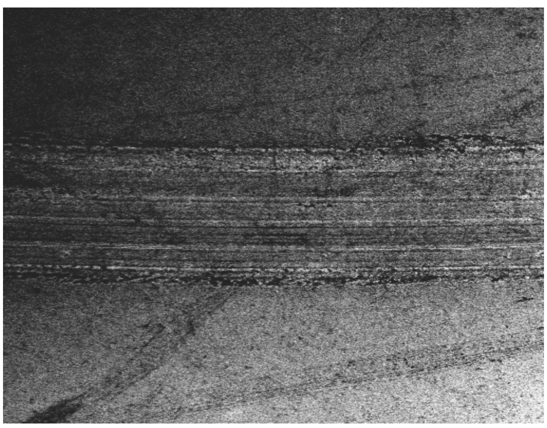


Fig. 12. Microphotography of wear track of the CrN coating, 20°C

4. Conclusions

1. The deposited PVD coatings onto the plasma nitrided X37CrMoV5-1 steel substrate are characterized by an identical thickness as well as a tight adhesion to the substrate material and a close construction without noticeable discontinuities in the form of delaminations or pores.
2. The structure of TiN/(Ti,Al)N coatings is columnar while of the CrN coating - fine-crystalline.
3. There is the friction coefficient connected with the roughness which, in turn, influences the resistance wear of the examined coatings. The smaller roughness, the smaller friction coefficient what leads to the increase of resistance wear of the examined coatings.
4. In case of some coatings the friction coefficient is characterized by the jumping course of changes. In case of the plasma nitride coatings the friction coefficient is influenced by the hardness and adhesion of the coatings. With the increase of the hardness of the PVD coatings there is the increase of the wear resistance as well.
5. The hardness of the investigated coatings is between 2443 $HV_{0,001}$ for the CrN coatings and 3354 $HV_{0,001}$ for the TiN/(Ti,Al)N coatings. The hardness of the examined PVD coating correlates with their adhesion to the substrate material.
6. All the examined PVD coatings the failures begin with the double-sided chippings on the scratch edges and flakes on their bottoms. These damages appear at different loadings depending on the type of the coating.
7. The increased hardness and resistance of the substrate in the plasma nitride layer contributes to the limitation of the fragmentation of coatings as a result of plastic deformation of the substrate, its conformal cracking, stratification, crumbling and delamination, what finally causes the increase of the adhesion parameters as a consequence of the scratch test.
8. A very good adhesion of the TiN/(Ti,Al)N coating to the plasma nitride steel substrate and its high hardness are connected with the good results of the pin-on-disc tribological test for this coating.
9. The type of the damages of the coating and the substrate, arisen during the scratch test, is similar to the damages and the character of wear during the tribological test.
10. It has been stated that the biggest resistance to the wear resistance at 20 and 500°C temperatures is characterized by the TiN/(Ti,Al)N coating, while the smallest resistance shows the CrN coating.

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