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The properties and wear resistance of the CrN PVD coatings

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Properties

ABSTRACT

Purpose: The aim of the paper is comparison of the mechanical properties and wear resistance of the monolayer CrN PVD coatings deposited onto heat treated and plasma nitrited X37CrMoV5-1 type hot work tool steel.

Design/methodology/approach: The microhardness tests of the PVD coatings were made on the dynamic ultra-microhardness tester. The surfaces' thopography and the structure of the PVD coatings was observed on the scanning electron microscopy. The evaluation of the adhesion of coatings to the substrate was made using the scratch test. The wear and friction tests were performed on a pin-on-disc device at the room temperature and at the temperature of 500°C. The friction coefficient between the ball and disc was measured during the test.

Findings: : In case of the CrN coating deposited onto the X37CrMoV5-1 nitrided hot work steel a very good adhesion has been revealed to the substrate material in comparison to the CrN coating deposited onto the heat treated hot work steel. Taking into account the results of measurements, one can state that the lowest wear at certain conditions at both room and elevated temperatures displays the CrN coating deposited onto plasma nitrited X37CrMo V5-1 hot work steel type.

Practical implications: The investigation results will provide useful information to applying the plasma nitriding and the CrN PVD coatings for the improvement of the wear resistance of tools made from hot work steels.

Originality/value: The paper contributes to better understanding of the structure, mechanical properties, adhesion and the wear resistance at the elevated temperature (500°C) of the monolayer CrN PVD coating deposited onto the heat treated and plasma nitrited hot work tool steel.

Keywords: Coatings; Structure; Adhesion; Wear resistance; Plasma nitriding

1. Introduction

The dynamical development of the industry poses higher and higher demands, especially in the area of the adjustment of their properties to the anticipated their operation conditions and applications. This regards, first of all, constructional materials, tool materials, copper alloys, etc. Achievement of the required high working properties is connected in most cases with the necessity of improvement of their manufacturing technologies, employment of novel manufacturing technologies or else modifications of the surface layer. 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, 2, 10, 13]. 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.

It has been documented in the literature that PVD coatings can reduce friction in tribological contacts and increase the abrasive wear resistance. However, application of PVD hard coatings to the relatively soft substrate cannot guarantee the optimal tribological performance. Their tribological performance is often limited by elastic and plastic deformation of the substrate, which can result in eventual coating failure [6, 7, 14, 15]. The best results in protection of tools made of hot work steel were obtained with duplex treatment procedure. Duplex treatments consisting of plasma nitriding and hard PVD coating have been proven to be successful in improving wear, fatigue and corrosion resistance of hot work tool steel substrate. By increasing the hardness of substrates, the nitrided case often provides a suitable load support for PVD coatings so that superior mechanical and tribological performance can be achieved. 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 [3-5, 9, 12]. The duplex surface treatment of the hot work tool steel for tools made for work at the elevated temperature improves their abrasion wear resistance significantly, compared to coatings developed with the PVD processes [8, 11].

The paper presents the results of the project focused on the investigation of the structure, adhesion and antiwear properties of CrN PVD coatings deposited onto heat treated and plasma nitrided hot work steel X37CrMoV5-1 type.

2. Material and method

The CrN coating was 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 heat treatment, the samples were ground and polished and the PVD coating was deposited. After the heat treatment 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, then the PVD coatings were deposited. The monolayer CrN coating was prepared in BALZERS BAI 730 deposition system by ion plating PVD process at 450°C temperature. The thickness of the investigated CrN coating deposited onto heat treated and plasma nitrited hot work tool steel measured using the kalotest method is 2.23 µm. A steel ball with a diameter of 11 mm is used in this method to develop a crater. The thickness measurements were made using the light microscope with the scale graduation. Ten measurements were made for each of the examined specimens to obtain the average values.

The surface roughness of the polished specimens and roughness of the PVD coatings were measured on the Taylor-Hobson Form Talysurf Series 2 profilometer. 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 the load of 0.981 N. Hardness tests of the investigated PVD coatings were made using Vickers micro-hardness testing method. The surfaces' topography of the investigated PVD coatings was observed on the scaning 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 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 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. 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 temperature of 500°C was used to better understanding of the wear resistance at the elevated temperature of investigation PVD coatings deposition onto hot work tool steel X37CrMoV5-1 type. 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 –10 mm
- 7500 revolutions – 20°C – friction radius – 13 mm
- 1000 revolutions – 500°C – friction radius – 16 mm
- 7500 revolutions – 500°C – friction radius – 17.5 mm

Examinations of wear traces developed during the pin-on-disc test were made on the scaning microscope. Wear trace profiles were measured on the Taylor – Hobson Form Talysurf 120L laser profilometer in eight directions (every 45°).

3. Results and discussion

The morphology of the investigated PVD coatings deposited onto heat treated and 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 onto plasma nitrited hot work tool steel (Fig. 1a) when the presence in CrN coating onto heat treated hot work tool steel (Fig. 1b) was the smallest one. The results of this investigation correspond with the results of roughness and value of the friction coefficient. Metallographic examinations of coatings fractures show that CrN coating onto heat treated and plasma nitrited X37CrMoV5-1 steel type has a compacted submicrocrystalline structure and are characterised by a uniform thickness (Fig. 2a, b).

Roughness of the investigated PVD coatings is within the 0.09 – 0.175 µm range. Results of these examinations correspond with the metallographic test results. The results of these measurements correspond with the metallographic examinations made on the SEM. The topography of the coatings influences the a)

b)

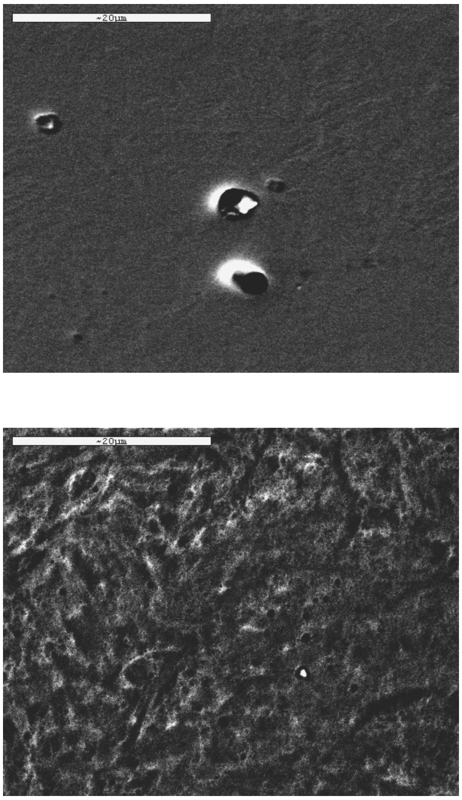


Fig. 1. Topography of the CrN coatings deposited onto hot work steel X37CrMoV5-1 type: a) after heat treated, b) after plasma nitrited

a)

b)

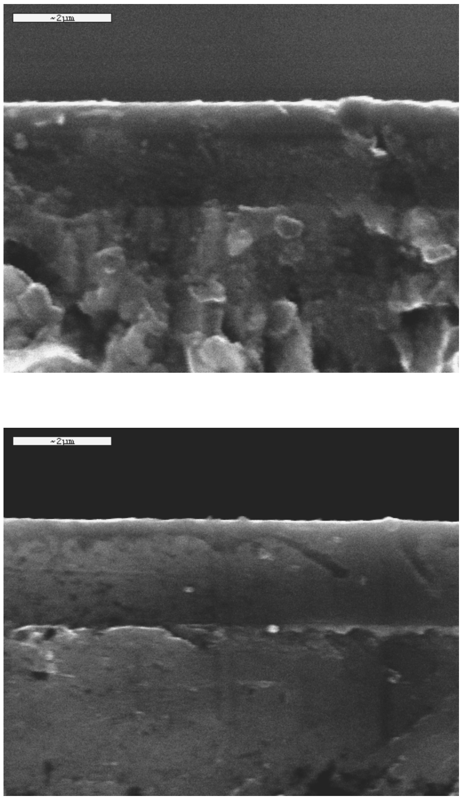


Fig. 2. Fracture of the CrN coatings deposited onto hot work steel X37CrMoV5-1 type: a) after heat treated, b) after plasma nitrited

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 CrN coatings onto plasma nitrited X37CrMoV5-1 type steel. The microhardness tests of the PVD coatings were carried out at 10 mN load, which ensures the limited indenter penetration depth to eliminate the substrate influence. The highest microhardness of 2443 $HV_{0.001}$ is characteristic of the CrN coating deposited onto plasma nitrited steel, and the lowest of 2410 $HV_{0.001}$ of the same coating onto heat treated hot work tool steel. The results of this investigation can be indicated the correlation between hardness and adhesion of the PVD coatings to the substrate materials.

It has been found out, on the basis of on the determined L_c (AE) values and on the developed failures metallographic examinations that monolayer CrN coating has very good adhesion to the substrate from the nitrided hot work tool steel, whereas the CrN coatings adhesion to the substrate from the heat treated hot work tool steel reaches the lowest value. The damage of the coatings commences in all cases with the widespread coating spalling on both edges of the originating scratch. The difference consists in the location of these spalling defects. In case of the CrN coating deposited onto plasma nitrided hot work tool steel the spalling defects begin at the load value of about 51 N (Fig. 3a-c).

Table 1.
Critical loads for CrN coating deposited onto heat treated and plasma nitrited X37CrMoV5-1 type hot work tool steel

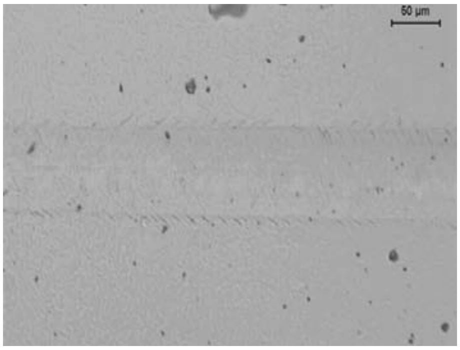
Substrate materials/coating type	Type of defect/Force [N]				
	L_c(AE)	L_{c3}	L_{c4}	L_{c5}	$L_c(F_t)$
X37CrMoV5-1+CrN	22.00	21.00	32.00	53.33	62.33
X37CrMoV5-1+PN+CrN	37.33	51.00	70.33	86.33	83.66

Next, cracks and coating stretches, develop on the scratch bottom, and finally the total coating delamination on the scratch bottom takes place. In case of the CrN coating deposited onto heat treated hot work tool steel the numerous spalling defects of the scratch edges begin at the load value of about 21 N. Spalling defects at the edges gets deeper and next coating delamination occurs (Fig. 4a-c).

The critical load values L_c, that are characterized by the adherence of the investigated PVD coatings to the substrate from the heat treated and nitrided hot work steel are presented in Table 1.

The CrN PVD coating on the plasma nitrided steel are characteristic of a better adhesion to the substrate material, compared to the adhesion of the same coatings 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. The maximum hardness of the 3 h long plasma nitrided layer onto the hot work tool steel X37CrMoV5-1 is 1478 $HV_{0,1}$ with the increase of the distance from the surface, microhardness (Fig. 5) of the investigated nitride layer goes down to 612 $HV_{0,1}$ specific for the core.

a)



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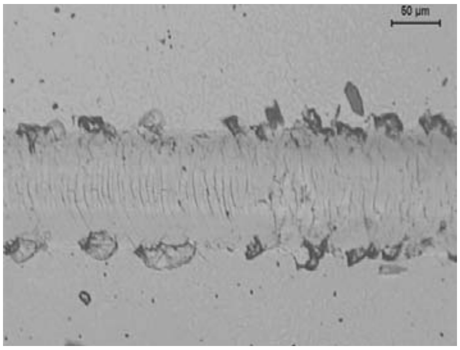


c)

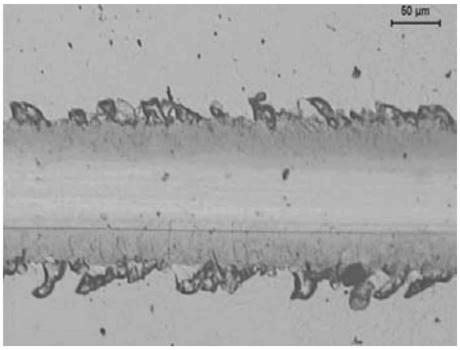
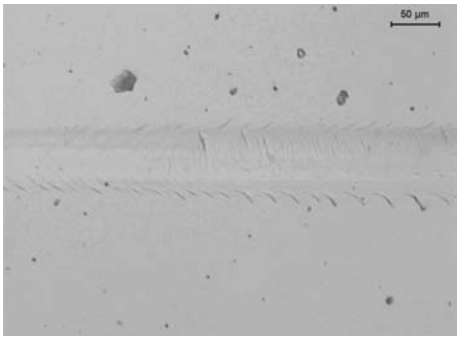


Fig. 3. Scratches with critical load: a) Lc_3-flaking on the scratch edge, b) Lc_4-partial delamination, c) Lc_5-total delamination; CrN coating deposited onto plasma nitrited X37CrMoV5-1 steel substrate

Test results of the investigated PVD coatings adhesion to the substrate from the heat treated and 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.

a)



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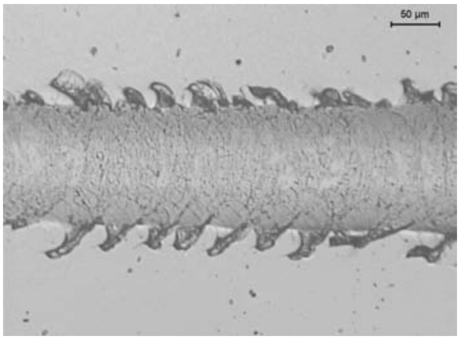


c)

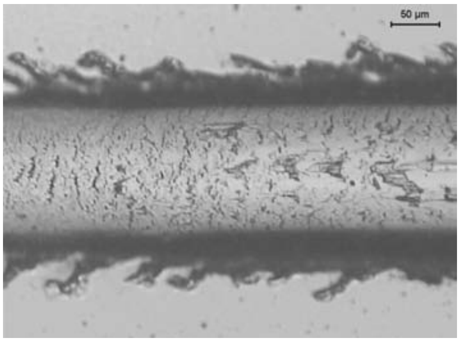


Fig. 4. Scratches with critical load: a) Lc_3-flaking on the scratch edge, b) Lc_4-partial delamination, c) Lc_5-total delamination; CrN coating deposited onto heat treated X37CrMoV5-1 steel substrate

Changes of the friction coefficient values between the corundum ball and the examined test piece were recorded during the tests. The analysis of the friction coefficient value changes of the investigated test pieces makes it possible to state that at the assumed experiment conditions the friction coefficient changes to about 0.6 for the CrN coating deposited onto nitrited hot work steel and to about 0.7 for the same coating onto heat treated hot work tool steel after 1000 test piece revolutions at the room temperature. The friction coefficient values for the coated test pieces are 0.5 for the CrN coating onto nitrited X37CrMoV5-1 steel type and 0.7 for the CrN onto heat treated steel at the same conditions of the test carried out at the temperature of 500°C. The increase of the test piece number of revolutions to 7500 at room temperature results in the change of values of the friction coefficients (Fig. 6a, b). However, one can state that they are close to the values obtained after 1000 revolutions at room temperature and are nearly the same – about 0.85 - for all examined coatings. The friction coefficient changes to 0.5 for the PN + CrN coating and to about 0.6 for the CrN one at the temperature of 500°C after 7500 revolutions (Fig. 7a, b).

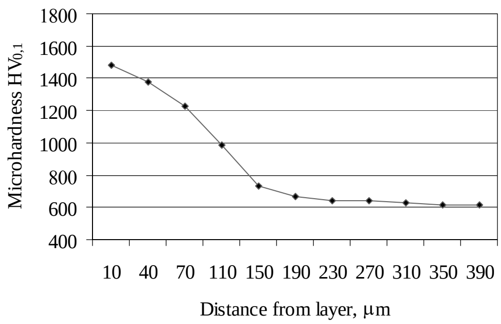
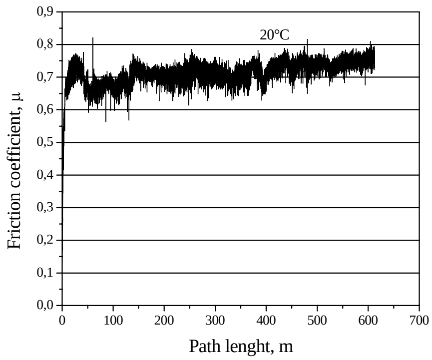


Fig. 5. Microhardness changes in the nitriding layer of the plasma nitrided hot work steel X37CrMoV5-1 type

a)



b)

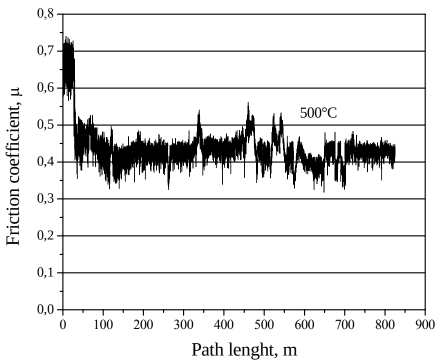


Fig. 6. Friction coefficient changes versus wear path length for CrN coating onto heat treated X37CrMoV5-1 steel type: a) 20°C, b) 500°C; 7500 revolutions

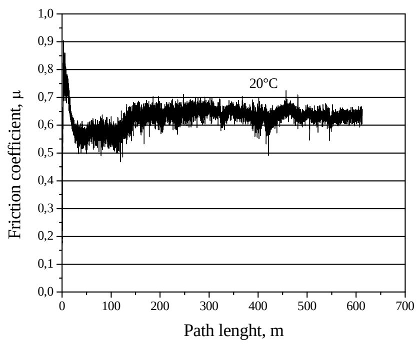
The CrN coating onto nitrited X37CrMoV5-1 steel type changes slightly its friction coefficient during the entire test period. The low values of the friction coefficient and their stable run are related to the adhesive character of the wear. The coating after the wear test at temperature of 20°C and 1000 and 7500 revolutions has had very little traces of wear. This tendency has also been observed after the test at the 500°C temperature. The strips and wear grooves developed in such conditions are very shallow. The quantitative evaluation of the examined test pieces surface wear due to friction was carried out based on the measurements of the scratch trace profiles on the CrN coatings put down onto the substrate from the heat treated and plasma nitrided X37CrMoV5-1 hot work steel and substrate material in eight directions every 45°. 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-disk 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 following formula:

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

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].

a)



b)

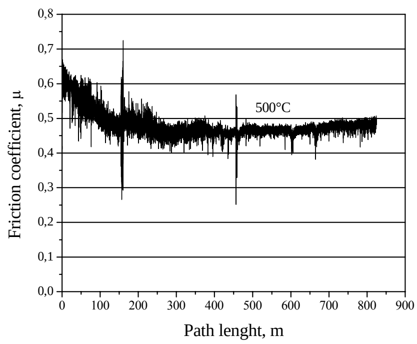


Fig. 7. Friction coefficient changes versus wear path length for CrN coating onto plasma nitrited X37CrMoV5-1 steel type: a) 20°C, b) 500°C; 7500 revolutions

a)

b)

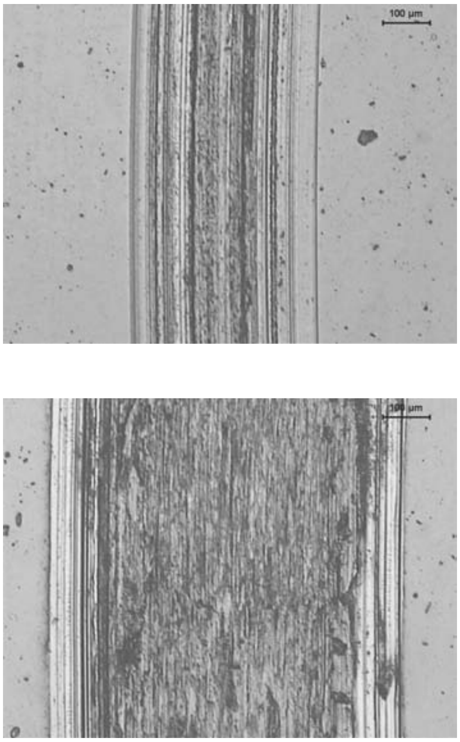


Fig. 8. Microphotography of wear track on the surface of investigated samples for CrN coating onto heat treated X37CrMoV5-1 steel type: a) 20°C, b) 500°C; 7500 revolutions

One can state, based 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 CrN coating onto plasma nitrited X37CrMoV5-1 steel type. The change of the number of revolutions from 1000 to 7500 causes the wear to increase threefold.

Evaluation results of the volume of material removed during the pin-on-disk test correspond with the wear trace width measurements made by observations carried out on the light microscope. The wear trace width values measured on the light microscope grow with the test temperature, regardless of the number of revolutions made by the test piece (Figs. 8a, b; 9a, b).

4. Conclusions

Based on the investigation results the following conclusions were arrived at:

- The deposited CrN PVD coatings onto heat treated and plasma nitrited 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. The structure of these coatings is compacted submicrocrystalline. What decides about the roughness of the PVD coatings deposited onto heat treated and plasma nitride steel is also the topography of the coatings with heterogeneous surfaces. The roughness of the investigated coatings is between R_a= 0.09-0.175 µm for the CrN onto heat treated X37CrMoV5-1 steel substrate and CrN coatings onto plasma nitrited steel adequately.
- The highest microhardness of 2443 $HV_{0.001}$ is characteristic of the CrN coating deposited onto plasma nitrited steel, and the lowest of 2410 $HV_{0.001}$ of the same coating onto heat treated hot work tool steel. The results of this investigation can be indicated the correlation between hardness and adhesion of the PVD coatings to the substrate material.
- The investigation monolayer CrN coating on the plasma nitrited steel is characteristic of a better adhesion to the substrate material (Lc_3=51 N), compared to the adhesion of the same coatings on the heat treated steel (Lc_3=21 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.

a)

b)

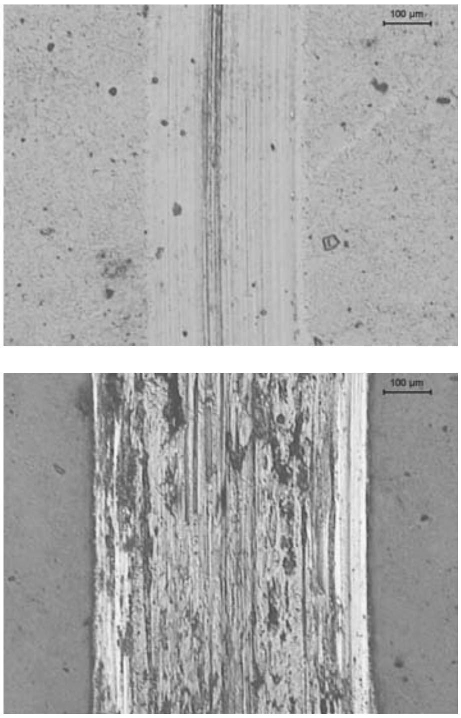


Fig. 9. Microphotography of wear track on the surface of investigated samples for CrN coating onto plasma nitrited X37CrMoV5-1 steel type: a) 20°C, b) 500°C; 7500 revolutions

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

Substrate materials/Coating type	Volume of materials removed V [mm^3]			
	1000 revolutions		7500 revolutions	
	20°C	500°C	20°C	500°C
X37CrMoV5-1+CrN	0.0356	0.1960	0.1475	1.1105
X37CrMoV5-1+PN+CrN	0.0262	0.1902	0.0654	0.7413

- It was found out that the deposition of coatings onto the plasma nitride steel there is the decrease of the friction coefficient values despite the increase of the roughness of these steels. In case of the plasma nitride coatings the friction coefficient is influenced by the hardness and adhesion of the coatings.
- It has been stated that the biggest resistance to the wear resistance at 20 and 500°C temperature is characterized by the CrN coating onto plasma nitrited X37CrMoV5-1 hot work tool steel, while the smallest resistance shows the same coating deposited onto head treated hot work tool steel.

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