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# Comparison of the adhesion and wear resistance of the PVD coatings

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**Properties**

## ABSTRACT

**Purpose:** of the paper was comparison of the adhesion and antiwear properties of the multilayer TiN/(Ti,Al)N PVD coatings deposited onto heat treated and plasma nitrited X37CrMoV5-1 type hot work tool steel.
**Design/methodology/approach:** 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 measured using Vickers micro-hardness testing method. The evaluation of the adhesion of coatings to the substrate was made using the scratch test. 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 friction coefficient between the ball and disc was measured during the test.
**Findings:** In case of the TiN/(Ti,Al)N coating deposited onto the X37CrMoV5-1 nitrided hot work steel show a very good adhesion which has been revealed to the substrate material is comparison to the TiN/(Ti,Al)N coating deposited onto heat treated hot work steel. Taking into account the results of measurements, one can state that the lowest wear at certain conditions in both room and elevated temperatures show TiN/(Ti,Al)N deposited onto plasma nitrited X37CrMo V5-1 hot work steel type.
**Practical implications:** The investigation results will provide useful information to applying of the TiN/(Ti,Al)N PVD coating for the improvement of wear resistance of tools made from hot work steels.
**Originality/value:** The paper contributes to better understanding the wear resistance at the elevated temperature to 500ºC of the multilayer TiN/(Ti,Al)N PVD coating deposited onto heat treated and plasma nitrited hot work tool steel
**Keywords:** Wear resistance; Adhesion; PVD coatings, Plasma nitriding

## 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. 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. Surface layers are obtained due to this process characteristic of the high hardness of 1200-1500HV, having also a good wear resistance. Plasma nitriding is carried out in the broad temperature range (400-590ºC), 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 [7-11]. 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 alloy hot-work tool steels. 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 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 [12-16].

The paper presents the results of the project focused on the investigation of adhesion and tribological properties of TiN/(Ti,Al)N PVD coatings deposited onto heat treated and plasma nitrided hot work steel X37CrMoV5-1 type. The investigation results will provide useful information to understanding and applying of these PVD coatings for the improvement of wear resistance of tools made from hot work steels.

## 2. Material and research methodology

The TiN/(Ti,Al)N coatings were deposited onto X37CrMoV5-1 type hot work steel substrate. The samples in the form of disc (diameter 55 mm and thickness 4 mm) were quenched at 1020˚C and tempered at 550˚C to hardness 55 HRC. After the thermal treatment, the samples were grounded and polished and the PVD coating was deposited. After the termal 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 - 3h, after nitiriding the samples were polished to a roughness $R_a$ = 0.08 µm, than the PVD coatings were deposited. The TiN/(Ti,Al)N multilayer coating was deposited by magnetron sputtering in CemeCon apparatus at the temperature 450˚C. The thickness of the TiN/(Ti,Al)N coating deposited onto heat treated and plasma nitrited hot work tool steel is 1.83 µm.

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. 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. 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 scaning microscope. Wear traces profiles were measured on the Taylor – Hobson Form Talysurf 120L laser profilometer in eight directions (every 45˚).

## 3. Results and discussion

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 tool steel, whereas the TiN/(Ti,Al)N 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 spallation on both edges of the originating scratch. The difference consists in the location of these spalling defects. In case of the TiN/(Ti,Al)N coating deposited onto plasma nitridited hot work tool steel the spalling defects begin at the load value of about 62 N. 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 TiN/(Ti,Al)N coating deposited onto heat treated hot work tool steel the spalling defects begin at the load value of about 45 N. Next there are flakes and conformal cracks connected with delamination. 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.

Table 1.
Critical loads for TiN/(Ti,Al)N coating deposited onto heat treated and plasma nitrited X37CrMoV5-1 type hot work tool steel

| Substrate material/coating type | Type of defect/Force [N] | | | | |
|---|---|---|---|---|---|
| | $L_c$(AE) | $L_{c3}$ | $L_{c4}$ | $L_{c5}$ | $L_c(F_t)$ |
| X37CrMoV5-1+TiN/(Ti,Al)N | 55.00 | 45.00 | 60.66 | 93.33 | 96.33 |
| X37CrMoV5-1+PN+TiN/(Ti,Al)N | 56.33 | 62.00 | 85.33 | 110.16 | 100.66 |

The TiN/(Ti,Al)N 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.

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. 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 0.4 for the TiN/(Ti,Al)N coating deposited onto nitrited hot work steel and to about 0.5 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 TiN/(Ti,Al)N coating onto nitrited X37CrMoV5-1 steel type and 0.7 for the TiN/(Ti,Al)N 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. 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 + TiN/(Ti,Al)N coating and to about 0.6 for the TiN/(Ti,Al)N one at the temperature of 500˚C after 7500 revolutions (Fig.1).

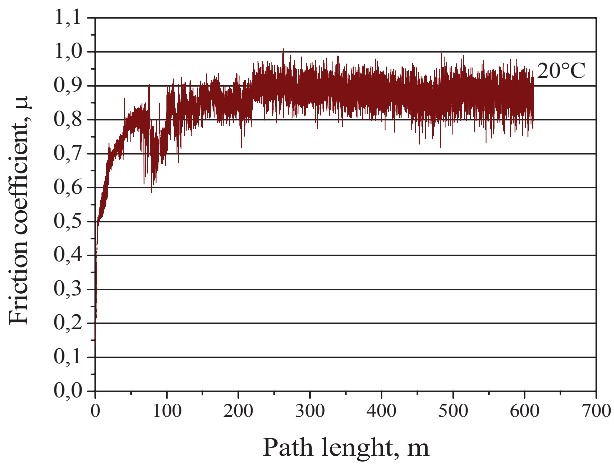


Fig.1. Friction coefficient changes versus wear track length for TiN/(Ti,Al)N coating onto plasma nitrited X37CrMoV5-1 steel type: 20˚C; 7500 revolutions

The TiN/(Ti,Al)N 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 arisen in such conditions strips and wear grooves are very shallow. The quantitative evaluation of the examined test pieces surface wear due to friction was carried out basing on the measurements of the scratch trace profiles on the TiN/(Ti,Al)N coatings put down onto the substrate from the heat terated 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:

$$V=(\pi*R*D^3)/(6*r) \quad (1)$$

where: V – average volume of the material worn out due to friction [$mm^3$], R – friction radius [mm], D – wear trace width [mm], r – ball radius [mm]

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 onto plasma nitrited X37CrMoV5-1 steel type. The change of the number of revolutions from 1000 to 7500 causes the wear to increase threefold.

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

| Substrate material/Coating type | Volume of materials removed V [$mm^3$] | | | |
|---|---|---|---|---|
| | 1000 revolutions | | 7500 revolutions | |
| | 20˚C | 500˚C | 20˚C | 500˚C |
| X37CrMoV5-1+TiN/(Ti,Al)N | 0.00691 | 0.19082 | 0.05043 | 0.36889 |
| X37CrMoV5-1+PN+TiN/(Ti,Al)N | 0.00516 | 0.07725 | 0.02515 | 0.32537 |

Wear test results for the investigated coatings correspond with the coating adhesion to the substrate material test results. The TiN/(Ti,Al)N coating onto plasma nitrited hot work tool steel 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 onto plasma nitrited hot work tool steel is characteristic of the best wear resistance compared to the same coating onto heat treated X37CrMoV5-1 steel type. 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 highest material wear at the temperature of 500°C. The deposition of TiN/(Ti,Al)N coating 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.

## 4. Conclusions

It was found out that the TiN/(Ti,Al)N PVD coating on the plasma nitrided X37CrMoV5-1 hot work tool steel is characteristic of a better adhesion ($L_{c3}$ = 62 N) to the substrate material, compared to the adhesion of the same coating on the heat treated steel ($L_{c3}$ = 45 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 weare resistance. One can state the highest friction coefficient of 0.6 to 0.7 show TiN/(Ti,Al)N coating deposited onto heat treated steel while the TiN/(Ti,Al)N coating, deposited onto plasma nitride steel at the 20 and 500°C temperatures show the lowest friction coefficient of 0.4-0.5. As a result of 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. A very good adhesion of the TiN/(Ti,Al)N coating to the plasma nitride steel substrate is connected with the good results of the pin-on-disc tribological test for this coating. 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. During this test the coatings are worn in the adhesion-abrasive way, and the damage, in most cases, reaches the material substrate. It has been stated that the biggest resistance to the wear resistance at 20 and 500°C temperature is characterized by the TiN/(Ti,Al)N 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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