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## Tribological Investigations of TiC+a-C:H Coatings Manufactured on X38CrMoV5-1 Steel Using PVD Technology

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**Keywords:** PVD method, Cathodic Arc Evaporation, TiC+a-C:H coatings, Tribological investigations, Pin-on-disc test, Wear resistance.

**Abstract.** X38CrMoV5-1 steel is a typical tool steel commonly used in forging and plastic moulding industry for production of ejectors, slides, dies, etc. In plastics moulding a lot of these parts sustain relative movement. Because of this, some seizing or micro-welding may appear, especially when lubrication is not used. For many years, the different types of protective coatings were developed to avoid such problems. Most of the obtained solutions relate to the manufacturing of low friction coatings obtained by different nitriding processes and by CVD or PVD methods. In this article, the friction coefficients and the wear resistances of TiC+a-C:H protective coatings manufactured on X38CrMoV5-1 steel samples by using PVD technology are studied. The investigations are based on tribometer tests in different temperature conditions. The process of deposition of PVD coatings was realized by using multisource, hybrid factory-scale equipment of type URM 079. This equipment allows for deposition of coatings by a physical method. The tribological tests were performed using a precision high temperature tribometer under ambient and high temperature conditions with a steel and corundum balls as a counter-samples. In this paper, the results of these tribological tests are presented. It is shown that the measured friction coefficient of steel samples with PVD coatings is significantly lower than the friction coefficient of uncoated steel. It is also shown that X38CrMoV5-1 steel samples with manufactured TiC+a-C:H coatings are characterized by a very low friction coefficient and high wear resistance.

### Introduction

X38CrMoV5-1 steel (ASTM designation) is a very commercial hot working steel characterized by very good mechanical properties (high tensile strength, hardness, high abrasion resistance, high yield stress – 2200 MPa). For this reason X38CrMoV5-1 steel is widely used in various branches of industry e.g. in die casting and plastic forming, cf. [1]. Moreover, its abrasive wear resistance and hardness (especially in the surface layer) can be significantly improved through thermochemical treatments or by application of surface coatings (nitriding, CVD and PVD coatings) as well as by laser alloying of the surface with tungsten carbide [2]. Steel producers try to outdo one another in developing specialized grades of steel and treatments for them. In this paper, the technology of manufacturing of TiC+a-C:H protective coatings on X38CrMoV5-1 steel using PAPVD (Plasma Activated Physical Vapour Deposition) method [3-5] is proposed. Moreover, the tribological investigation concerning measurement of friction coefficient and wear resistance of manufactured coatings is presented. For the investigations the X38CrMoV5-1 steel samples from LaBoMaP-Centre Arts et Métiers de Cluny-France were used. The chemical composition (in wt%) of investigated X38CrMoV5-1 steel is: 0.42% C, 0.5% Mn, 1.2% Si, 5.5% Cr, 1.4% Mo, 0.3% V, Fe balance. The series of samples of 30 mm diameter and 2 mm thickness were made. The main requirement of the preparation process of samples for deposition TiC+a-C:H protective coatings is to obtain the solid surface layer with low roughness and devoid of all impurities. To this end, the technological process of preparation of the samples is elaborated by using a precision grinder-polisher machine.

### Preparation of X38CrMoV5-1 steel samples for investigations

The polishing process of samples made of X38CrMoV5-1 steel was carried out using Phoenix Beta 2 (Buehler-Germany) dual platen grinder-polisher machine equipped with a Vector power head and applying Buehler abrasive materials and lubricants. Process stages of realized operations contained:

- two-stage grinding of samples using BuehlerMet silicon carbide (SiC) abrasive paper P600 (grits size  $\varnothing$  26  $\mu\text{m}$ ) and P1200 (grits size  $\varnothing$  15  $\mu\text{m}$ );
- lapping of samples using medium hard woven silk cloth VerduTex and monocrystalline diamond suspension MetaDi (grains size  $\varnothing$  3  $\mu\text{m}$ );
- polishing of samples using soft synthetic pad ChemoMet and aluminium oxide ( $\text{Al}_2\text{O}_3$ ) final polishing suspension MasterPrep (grains size  $\varnothing$  0,05  $\mu\text{m}$ ).

The elaborated technological process ensures suitable preparation of the steel samples in the range of required roughness parameters. It also ensures proper purity of the samples. The surface of polished samples had a silver glossy colour and invisible tool marks.

### Experimental apparatus and procedure

For deposition of TiC+a-C:H coatings on X38CrMoV5-1 circular steel samples the modified hybrid PVD Cathodic Arc Evaporation (PVD CAE) system was used. The process of manufacturing of these coatings was realized in the Institute of Materials Science at Technical University of Lodz by using multisource, hybrid factory-scale equipment of type URM 079. This equipment allows for deposition of coatings by a physical method, cf. [3-5]. It has two independent continuous arc power supplies with magnetic filtration of microdrop for production of metallic plasma (maximum discharge current 180 V), two pulsating carbon plasma power supplies (average pulse power 1 kW) and magnetron source (power 10 kW) with a flat circular target of 100 mm of dimension. This equipment allows for plasma cleaning of the surface by four independent ion guns (4 kW/100 mA) in argon atmosphere. In the range of high voltages the polarization voltage of substrate was controlled from 800-2500 V. In the range of low voltages it was controlled from 0-200 V. The nominal vacuum was below  $1,0 \times 10^{-4}$  Pa. The scheme of the vacuum chamber of PVD stand and its equipment is presented in Figure 1.

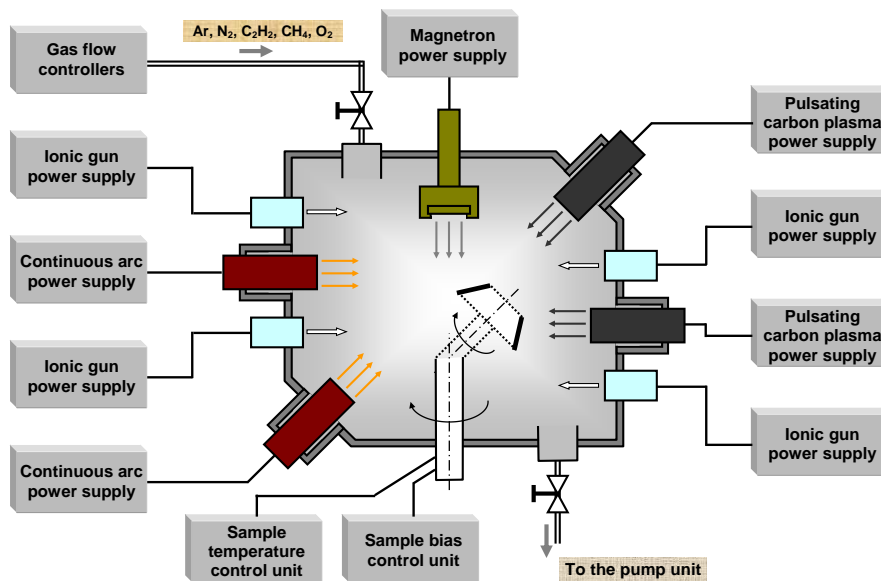


Fig. 1. Scheme of arrangement of separate parts in the vacuum chamber of PVD Cathodic Arc Evaporation stand used for deposition of TiC+a-C:H coatings.

### Microstructure of X38CrMoV5-1 steel samples with TiC+a-C:H coatings

For comparison of microstructure of uncoated X38CrMoV5-1 steel samples with microstructure of steel samples with TiC+a-C:H coatings the optical profiler WYKO NT 1100 (Veeco Instruments-USA) was used. The results of investigations are presented in Figure 2.

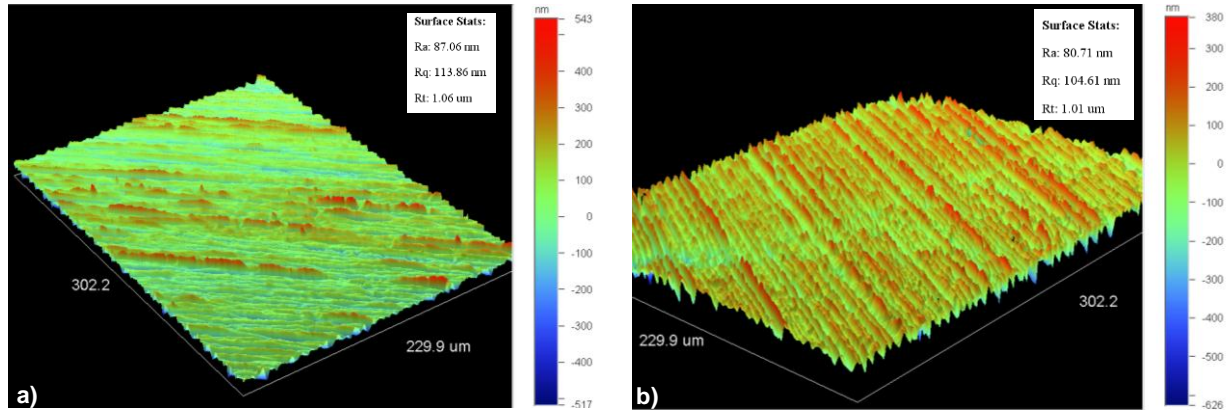


Fig. 2. Microstructure images of X38CrMoV5-1 steel samples: a) uncoated after polishing, b) with TiC+a-C:H coatings.

The results obtained here show that the surface layer of X38CrMoV5-1 steel samples with TiC+a-C:H coatings have a regular, smooth structure and lower average roughness parameters ( $R_a=0.08 \mu\text{m}$ ) than uncoated samples ( $R_a=0.087 \mu\text{m}$ ).

### Tribological investigations of TiC+a-C:H coatings manufactured on X38CrMoV5-1 steel samples

Tribological investigations of TiC+a-C:H coatings manufactured on X38CrMoV5-1 steel samples were done by using computer-controlled pin-on-disk tribometer (CSM Instruments-Switzerland) with a heated sample holder and with applying steel (100Cr6) and corundum ( $\text{Al}_2\text{O}_3$ ) balls as a counter-samples. Tribological tests were conducted not only at ambient conditions ( $25^\circ\text{C}$ ) but also at a temperature  $200^\circ\text{C}$  because in some cases, moulds are heated in this range of temperature. Parameters of the friction coefficient test were as follows: load - 5N, frequency - 5 Hz, counter-sample ball radius - 6 mm, average velocity - 10 cm/s, distance 200-800 m. The results of friction coefficient measurements of uncoated X38CrMoV5-1 steel samples at  $25^\circ\text{C}$  and  $200^\circ\text{C}$  with using counter-samples made of different materials are presented in Figs. 3-4. Comparison of friction measurements of X38CrMoV5-1 steel with TiC-a-C:H coating at  $25^\circ\text{C}$  and  $200^\circ\text{C}$  with using counter-samples made of different materials are shown in Fig. 5 and Fig. 6.

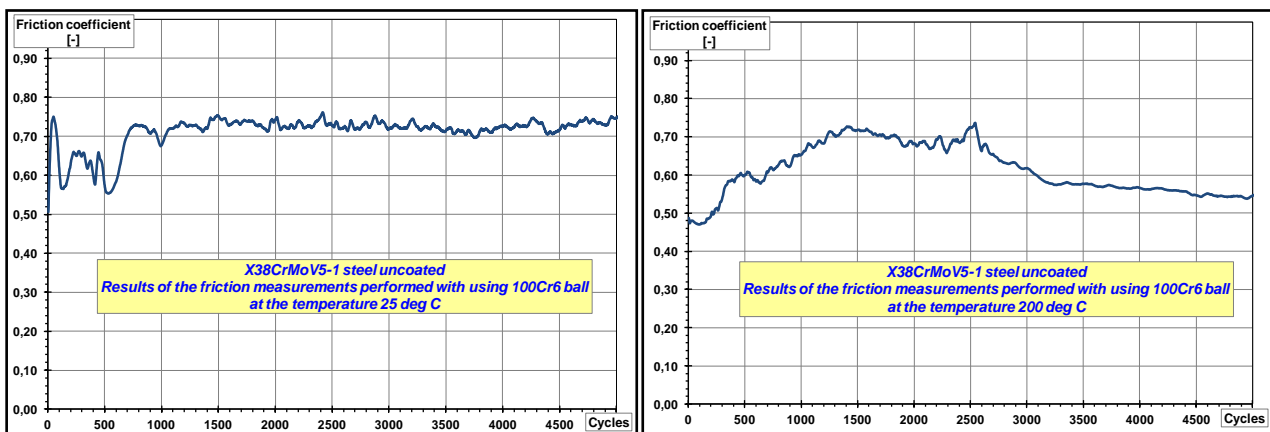


Fig. 3. Friction measurements of uncoated X38CrMoV5-1 steel conducted at the temperature  $25^\circ\text{C}$  and  $200^\circ\text{C}$  using 100Cr6 ball as a counter-sample.

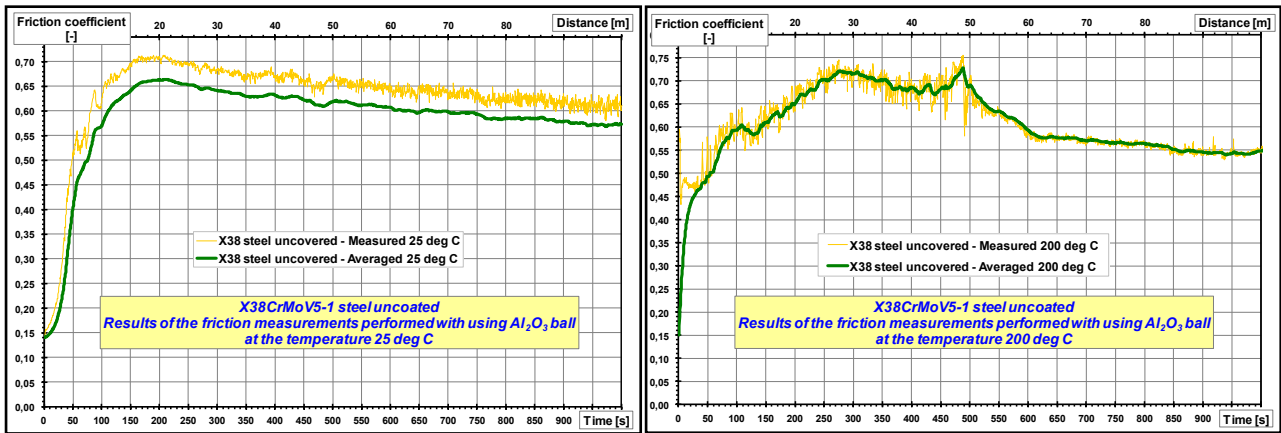


Fig. 4. Friction measurements of uncoated X38CrMoV5-1 steel conducted at the temperature 25°C and 200°C using Al<sub>2</sub>O<sub>3</sub> ball as a counter-sample.

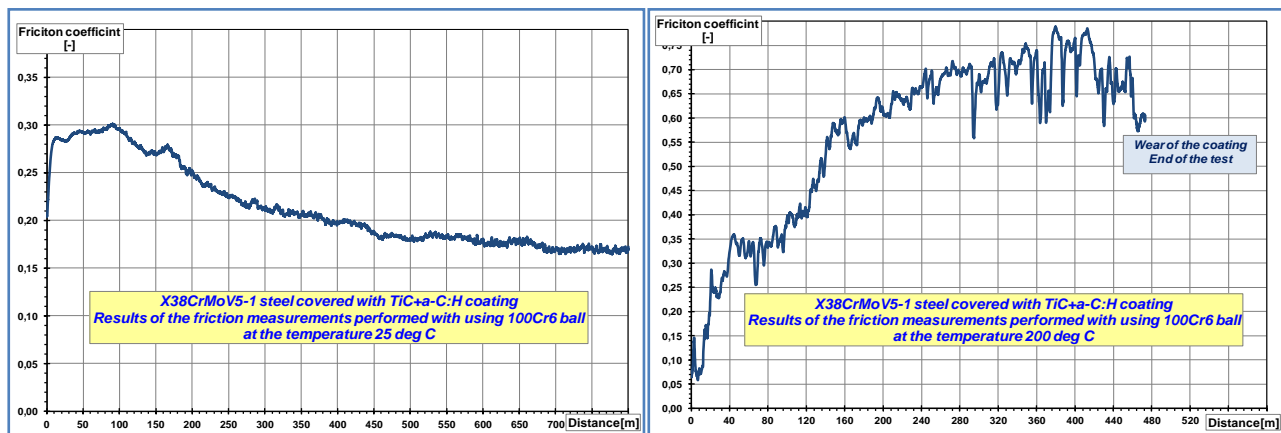


Fig. 5. Comparison of friction measurements of X38CrMoV5-1 steel with TiC-a-C:H coating conducted at the temperature 25°C and 200°C using 100Cr6 ball as a counter-sample.

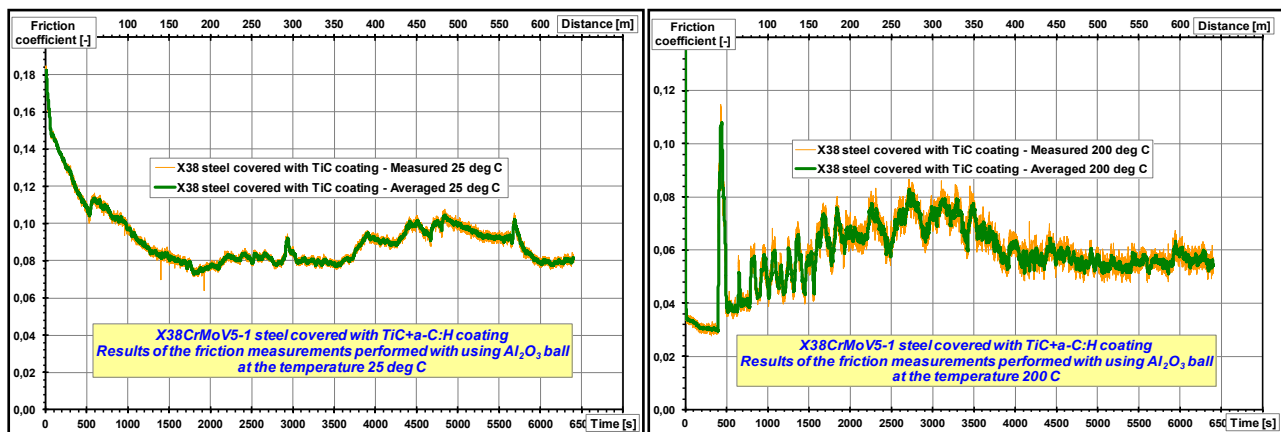


Fig. 6. Comparison of friction measurements of X38CrMoV5-1 steel with TiC-a-C:H coating conducted at the temperature 25°C and 200°C using Al<sub>2</sub>O<sub>3</sub> corundum ball as a counter-sample

From the results presented above it follows that the measured friction coefficient of X38CrMoV5-1 steel samples with manufactured TiC-a-C:H coatings is significantly lower as compared with the steel samples without these coatings, especially at ambient conditions (25°C). Moreover, it can be observed that the lowest values of friction coefficient both at 25°C and 200°C take place for corundum (Al<sub>2</sub>O<sub>3</sub>) ball as a counter-sample and even after a 800 meter test the friction coefficient does not increase (Fig. 6). It means that TiC-a-C:H coatings manufactured on X38CrMoV5-1 are characterized by very high wear resistance.

### Wear rate of TiC+a-C:H coatings manufactured on X38CrMoV5-1 steel

For investigations of wear tracks of TiC+a-C:H coatings manufactured on X38CrMoV5-1 steel samples the optical profilometry was done by using the WYKO NT 1100 optical profiler (Veeco Instruments-USA). It allows for obtaining the profile of the track. The coatings wear tracks look like circular grooves. With the cross-sectional area of the track taken on several points it is possible to calculate the worn volume of the coatings and then to calculate the wear rate. In order to obtain a representative average, for measurements we have chosen 8 sections, one at every 45°. One multiplies the average section by the perimeter to have the volume:

$$V_{track} = 2\pi \cdot r \cdot S \quad (1)$$

where  $V_{track}$  is the worn volume ( $m^3$ ),  $r$  is the track radius (mm) and  $S$  is the average section ( $mm^2$ ). The wear rates of the coating and ball are calculated according to the equation [6]:

$$K = \frac{V}{w \cdot s} \quad (2)$$

where:  $V$  is the worn volume ( $m^3$ ),  $w$  is the load (N) and  $s$  is the sliding distance (m).

Obtained results are depicted in Figures 7-8. However pictures of wear tracks of TiC+a-C:H coatings using optical microscopy are shown in Figures 9-10.

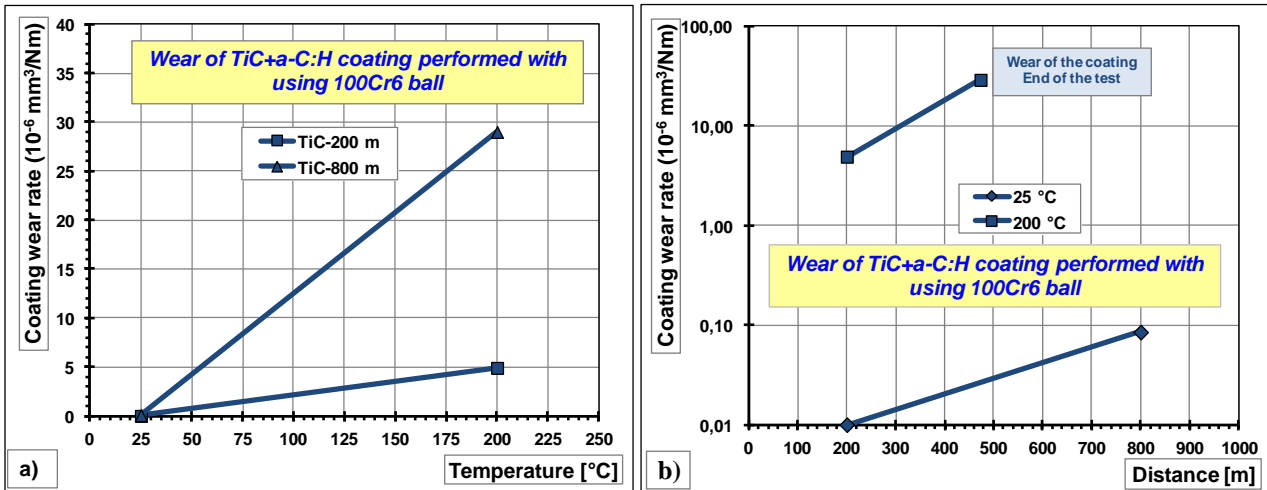


Fig. 7. The wear rate of the TiC+a-C:H coating manufactured on X38CrMoV5-1 steel with using 100Cr6 ball as a counter-sample: a) coating wear rate as a function of temperature, b) coating wear rate as a function of sliding distance.

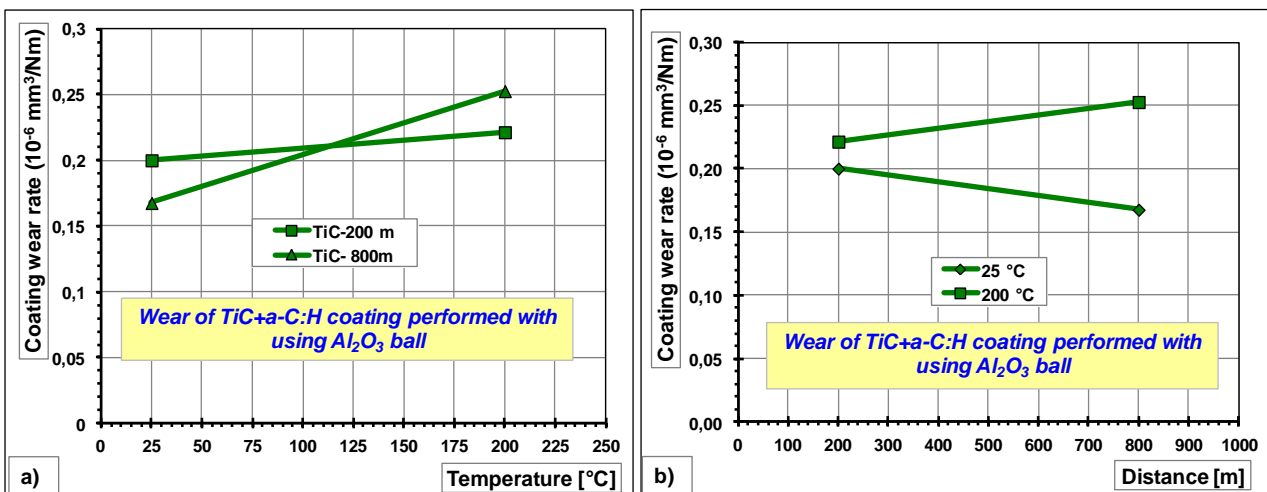


Fig. 8. The wear rate of the TiC+a-C:H coating manufactured on X38CrMoV5-1 steel with using  $Al_2O_3$  ball as a counter-sample: a) coating wear rate as a function of temperature, b) coating wear rate as a function of sliding distance.

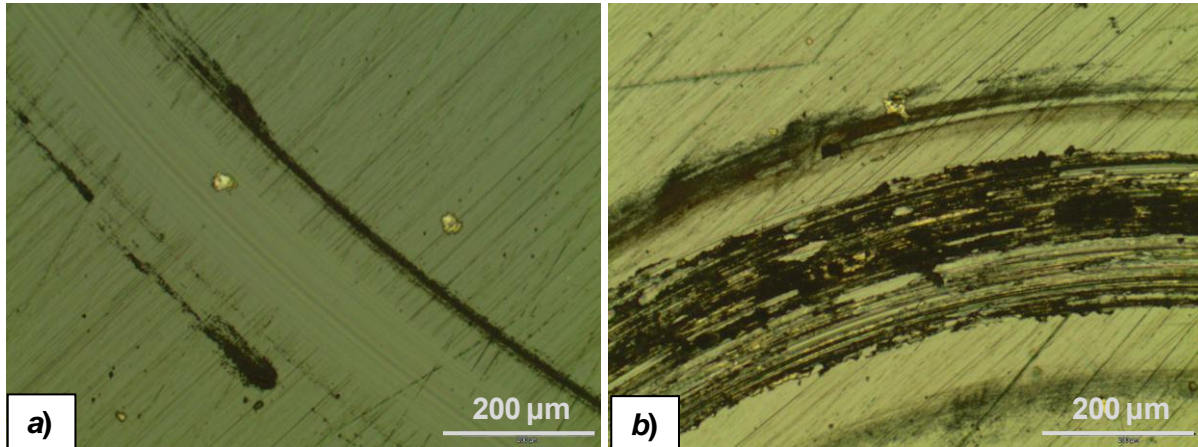


Fig. 9. Wear tracks of TiC+a-C:H coating manufactured on X38CrMoV5-1 steel sliding against 100Cr6 ball: a) after 800 meter test at 25°C, b) after 450 meter test at 200°C.

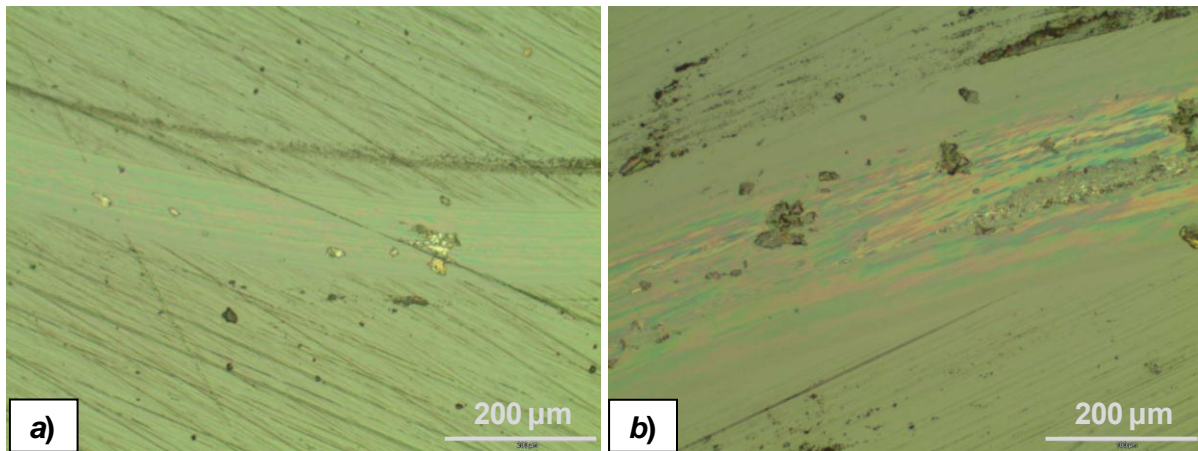


Fig. 10. Wear tracks of TiC+a-C:H coating manufactured on X38CrMoV5-1 steel sliding against Al<sub>2</sub>O<sub>3</sub> ball: a) after 800 meter test at 25°C, b) after 800 meter test at 200°C.

The wear rate of TiC+a-C:H coating with using 100Cr6 ball as a counter-sample at the temperature 25°C and after 800 meter test is very low ( $0.085 \times 10^{-6}$  mm<sup>3</sup>/Nm). However, at the temperature 200°C the wear rate rapidly increases to  $2.899 \times 10^{-5}$  mm<sup>3</sup>/Nm after the 450 meter test (Fig. 7 and Fig. 9). The wear rate of TiC+a-C:H coating with using Al<sub>2</sub>O<sub>3</sub> ball as a counter-sample at the temperature 25°C and 200°C as well as after the 800 meter test is very low and its averaged values are respectively  $0.167 \times 10^{-7}$  mm<sup>3</sup>/Nm and  $0.252 \times 10^{-7}$  mm<sup>3</sup>/Nm (Fig. 8 and Fig. 10).

### Wear rate of the counter-samples made of 100Cr6 and Al<sub>2</sub>O<sub>3</sub> balls

The investigations of the wear rate of the counter-samples were done basing on the morphology of the balls scars with using optical microscope. The morphology of the balls scars gives information about the friction mode and the wear mode. The size of the scar allows for calculating the ball wear rate. The worn material of the balls is similar to a spherical cap.

The worn volume of the balls is calculated according to the equations [7]:

$$h_{cap} = R_{ball} - (R_{ball}^2 - r_{scar}^2)^{\frac{1}{2}} \quad (3)$$

$$V_{cap} = \frac{\pi \cdot h_{cap} \cdot (h_{cap}^2 + 3 \cdot r_{scar}^2)}{6} \text{ or } V_{cap} = \frac{\pi \cdot h_{cap}^2 \cdot (3 \cdot R_{ball} - h_{cap})}{3} \quad (4)$$

where:  $V_{cap}$  is the worn volume (m<sup>3</sup>),  $h_{cap}$  is the height of the cap (mm),  $R_{ball}$  is the radius of the ball (mm) and  $r_{scar}$  is the radius of the scar (mm).



Obtained results are presented in Figures 11-12. However pictures of wear tracks of 100Cr6 and Al<sub>2</sub>O<sub>3</sub> balls using optical microscopy are shown in Figures 13-14.

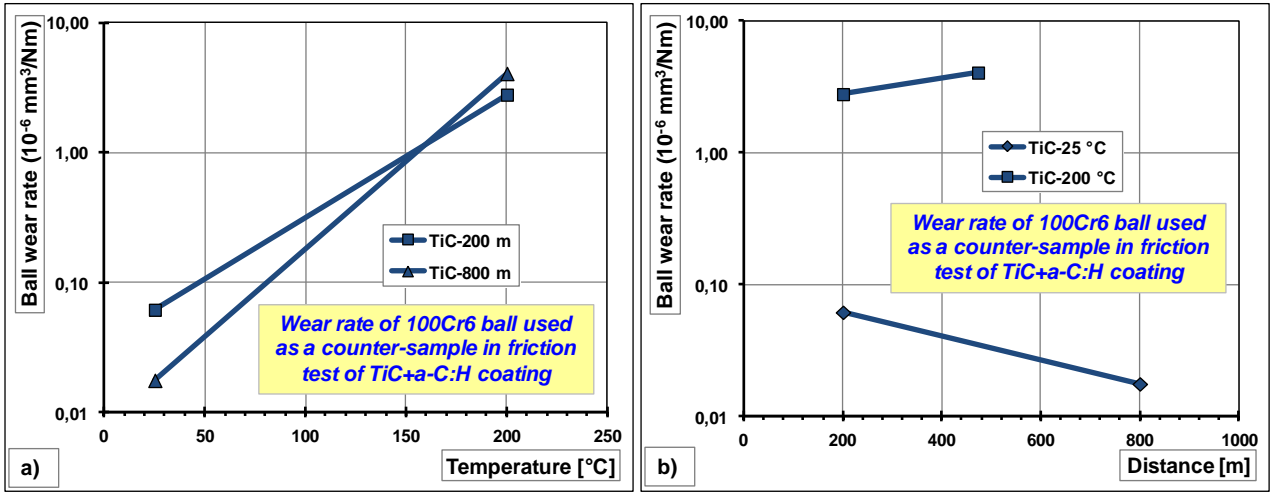


Fig. 11. The wear rate of 100Cr6 ball used as a counter-sample: a) ball wear rate as a function of temperature, b) ball wear rate as a function of sliding distance.

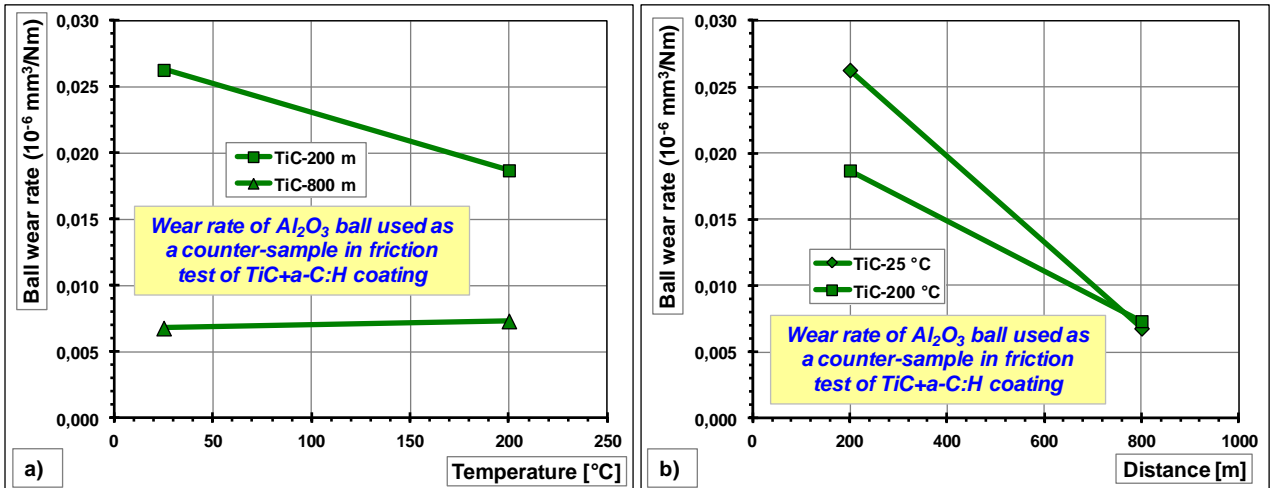


Fig. 12. The wear rate of Al<sub>2</sub>O<sub>3</sub> ball used as a counter-sample: a) ball wear rate as a function of temperature, b) ball wear rate as a function of sliding distance.

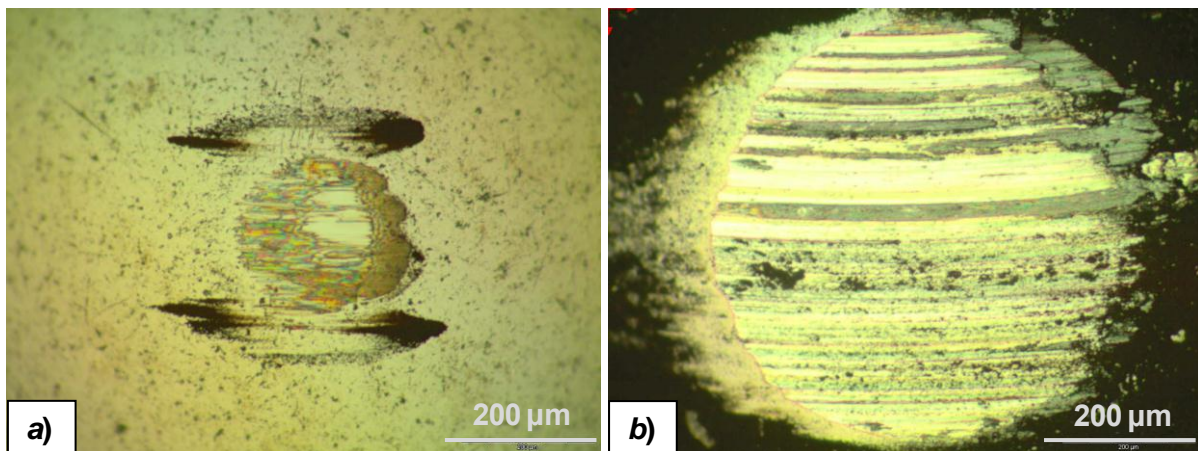


Fig. 13. Wear scar of 100Cr6 steel ball sliding against TiC+a-C:H coating manufactured on X38CrMoV5-1 steel: a) after 800 meter test at 25°C, b) after 450 meter test at 200°C.

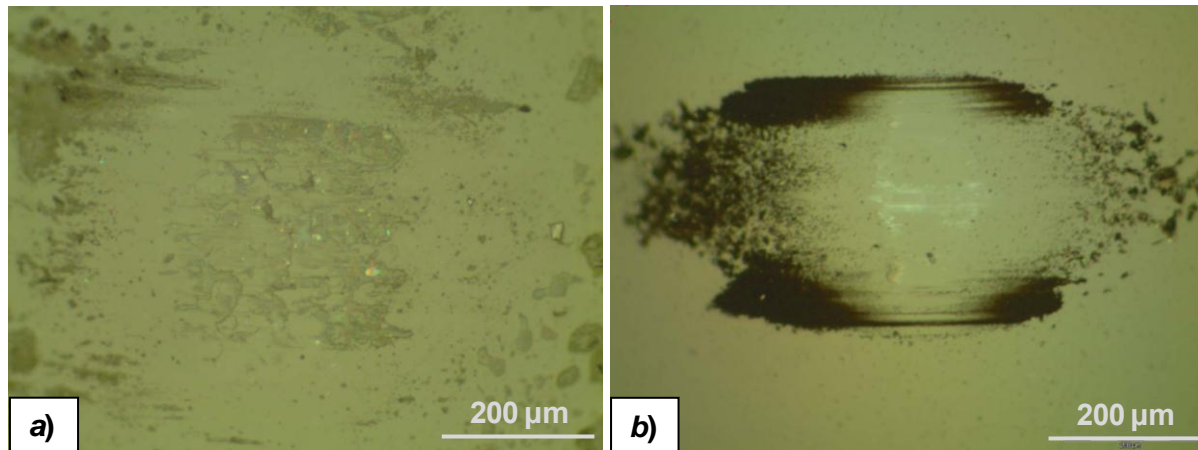


Fig. 14. Wear scar of  $\text{Al}_2\text{O}_3$  corundum ball sliding against TiC+a-C:H coating manufactured on X38CrMoV5-1 steel: a) after 800 meter test at  $25^\circ\text{C}$ , b) after 800 meter test at  $200^\circ\text{C}$ .

The wear rate of 100Cr6 ball used as a counter-sample in friction test of TiC+a-C:H coating at temperature  $25^\circ\text{C}$  is very small after the 200 meter test ( $6.206 \times 10^{-8} \text{ mm}^3/\text{Nm}$ ) and after the 800 meter test decreases even to  $1.777 \times 10^{-8} \text{ mm}^3/\text{Nm}$ . It means that the 100Cr6 ball probably wears during the running-in and acting at the same time like a polishing leveled the roughness of the coating. Since the surface of the ball and the coating are very smooth, the friction coefficient decreases and the wear slows down. However at the temperature  $200^\circ\text{C}$  the wear rate of 100Cr6 ball rapidly increases to  $4.080 \times 10^{-6} \text{ mm}^3/\text{Nm}$  after 450 meter test (Fig. 11 and Fig. 13). The wear rate of  $\text{Al}_2\text{O}_3$  ball used as a counter-sample at the temperature  $25^\circ\text{C}$  is also very small after the 200 meter test ( $2.628 \times 10^{-8} \text{ mm}^3/\text{Nm}$ ) and after the 800 meter test it decreases to  $6.790 \times 10^{-9} \text{ mm}^3/\text{Nm}$ . At the temperature  $200^\circ\text{C}$  the wear rate of  $\text{Al}_2\text{O}_3$  ball also decreases. After the 200 meter test it is equal to  $1.870 \times 10^{-8} \text{ mm}^3/\text{Nm}$ , however after the 800 meter test it is equal to  $7.870 \times 10^{-9} \text{ mm}^3/\text{Nm}$  (Fig. 12 and Fig. 14).

### Final remarks and conclusions

Investigations carried out in this paper have confirmed that PVD CAE method enables deposition of protective TiC+a-C:H coatings on X38CrMoV5-1 steel samples. Elaborated technological polishing process of X38CrMoV 5-1 steel samples ensures suitable preparation of their surfaces in the range of required roughness parameters. It also ensures proper purity of the samples. From the results it evidently follows that TiC+a-C:H coatings deposited on X38CrMoV5-1 steel samples considerably improve their tribological properties. It was shown that the measured friction coefficient of steel samples with PVD coatings is significantly lower than the friction coefficient of uncoated steel especially with using  $\text{Al}_2\text{O}_3$  corundum ball as a counter-sample. Wear tracks of TiC+a-C:H coating sliding against corundum ball are very small even after a 800 meter test and the wear of the coating is not observed. It means that TiC+a-C:H coatings are characterized by a very high wear resistance.

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