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FRICTION AND WEAR OF LOW TEMPERATURE DEPOSITED TIN COATING SLIDING IN DRY CONDITIONS AT VARIOUS SPEEDS

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Original scientific paper

In this research the influence of sliding speed on friction and wear of TiN coatings during dry reciprocating sliding against alumina ball was studied. Additionally, in order to find the optimum sliding speed, the coatings with different surface roughness were tested. The range of surface roughness analysed in this research provides insight into a rarely studied domain of fine surface finish. The wear tests were conducted with low load and low sliding speeds in order to determine the wear behaviour of TiN coatings in mild conditions. Low values of friction factor were obtained, ranging from 0,13 to 0,24. For all surface roughness the decreasing trend of the friction factor and increasing trend of the wear rate with increase in the sliding speed was observed. The optimum balance of low friction factor and the lowest wear rate was found for the coating with average roughness (*Ra*) value of 20 nm.

Keywords: *AFM, IBAD, reciprocating sliding, sliding speed, surface roughness, TiN*

Trenje i trošenje nisko temperaturno nanesene TiN prevlake klizanjem u suhim uvjetima pri različitim brzinama

Izvorni znanstveni članak

U radu su opisana provedena istraživanja utjecaja brzine klizanja na trenje i trošenje TiN prevlaka tijekom klizanja amo-tamo aluminijske kuglice. Pored toga, kako bi se utvrdila optimalna brzina klizanja, ispitivane su prevlake različitih hrapavosti. Istraživanjima obuhvaćeni raspon hrapavosti daje uvid u rijetko istraživano područje fine završne obrade. Pokusi trošenja su provedeni s malim opterećenjima i malim brzinama klizanja kako bi se utvrdilo ponašanje TiN prevlaka pri umjerenim opterećenjima. Utvrđene su niske vrijednosti faktora trenja u rasponu od 0,13 do 0,24. Kod svih hrapavosti opažen je trend opadanja faktora trenja i trend povećanja brzine trošenja s povećanjem brzine klizanja. Optimum niskog faktora trenja i niske brzine trošenja je utvrđen pri srednjoj hrapavosti (*Ra*) od 20 nm.

Ključne riječi: *AFM, brzina klizanja, IBAD, klizanje amo-tamo, površinska hrapavost, TiN*

1 Introduction

Industrial application of nitride based hard coatings on steel is steadily expanding. High hardness, low friction factor, good adhesion to steel substrates and good thermal and chemical stability make TiN coatings suitable for wear protection of various mechanical components. Although tribological properties of TiN coatings have been widely studied, there is still a number of possible application areas where behaviour of TiN coatings during wear is to be investigated. Understanding of wear mechanisms occurring during contact of TiN with other materials plays a great role in expanding its exploitation field [1].

Great number of parameters can influence the tribological behaviour of coated mechanical components. Tribological behaviour of selected coating depends on: (1) mechanical, physical and chemical properties of counter material; (2) surface roughness and (3) working conditions such as environment, contact geometry or contact pressure. In practice, it is not possible to theoretically link these factors to tribological response of coated elements. True tribological behaviour can only be determined experimentally [2 ÷ 4]. Tribological studies of hard coatings are usually conducted by using sliding test with high normal loads and high sliding speeds. On the other hand, mechanical components often wear out at rates of nanometres per hour. In addition, a number of applications where sliding occurs at low normal loads and low sliding speeds is constantly increasing [5, 6]. In order to evaluate the coating tribological properties for the above mentioned wear conditions, reciprocating sliding under milli-Newton loads was used in this research. Special consideration was given to the influence of sliding speed on the friction and wear of coatings with different surface roughness. There are several parameters which are applied to quantify the wear performances of hard coatings. Although a worn volume is the one usually used, it is not as practical as a wear rate [7]. Therefore, in the present study the wear rate is used to quantify the coating wear performance.

2 Materials and experimental

Studied coatings were prepared in an Ion Beam Assisted Deposition (IBAD) chamber with a base pressure of $1,5 \times 10–6$ mbar. In order to enhance the coating-substrate adhesion the substrates were etched by argon ions for 15 min before the coating deposition. Ion Beam Mixing Technique was applied to produce a 30 nm thick Ti interlayer which was followed by the deposition of TiN coating. During all stages of the deposition process the substrates temperature did not exceed 50 °C. As a substrate material a carburizing steel (20MnCr5) disks were used. Selected steel is commonly used for highly stressed components like gears and crankshafts. To reproduce a real application condition steel disks were carburized and quenched. Variation in surface roughness was obtained by the following methods: Specimen 1 - substrate ground using 400 grit SiC paper; Specimen 2 - substrate ground using 1500 grit SiC paper; Specimen 3 - substrate polished using 1μm diamond paste.

Ball cratering was utilized in order to produce a depression with the shape of a spherical cap. Measured dimensions of the depression were used for the calculation of the coating thickness. Substrate hardness was measured by standard Vickers hardness tester. On the other hand, the coating hardness was assessed using the

"Fischerscope HM2000 S" Microhardness Measurement System by applying maximum load of 3 mN in order not to exceed the 10 % of the coating thickness during indentation.

Tribological characterization was performed by ball-on-plate tribometer. Dry reciprocating sliding tests were conducted in air at room temperature. An alumina ball with diameter of 1,5 mm was used as a counterpart material. The wear tests were conducted using the sliding speeds of 10, 15 and 25 mm/s with applied normal load of 100 mN. A stroke length of 1mm was used in all tests which were stopped after 3000 cycles. All tribo-tests were repeated two times.

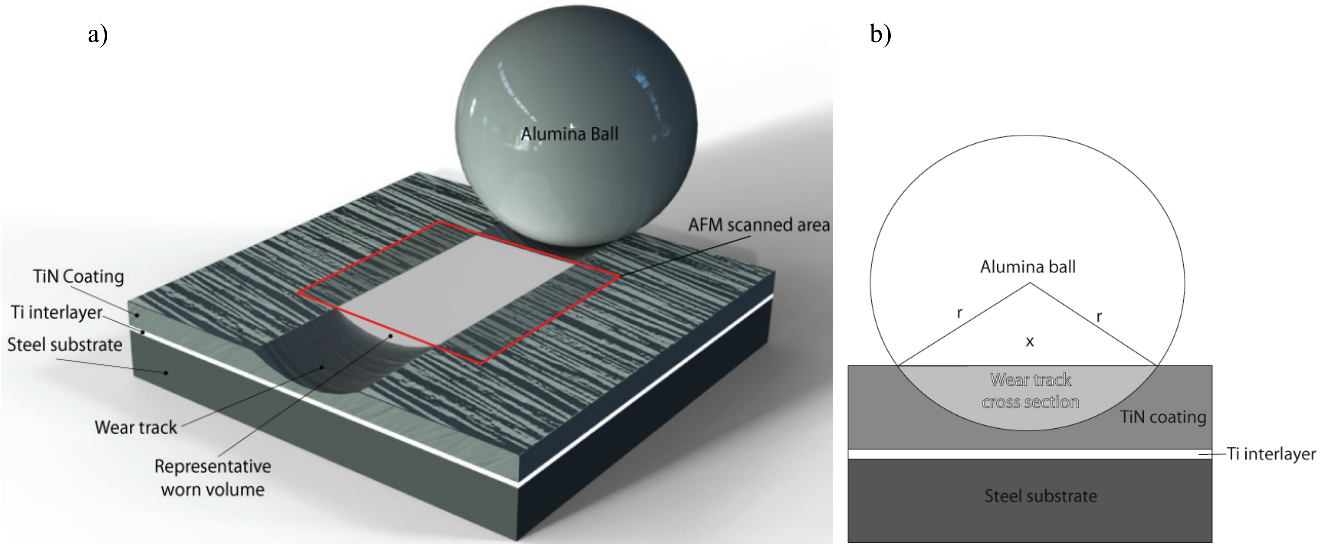

Figure 1 Schematic illustrations: a) tribo-contact and AFM measuring method; b) geometry used for calculation

Scanning electron microscope (SEM JEOL JSM 6460 LV with an embedded Oxford Instrument EDX analyser) was applied to investigate the morphology and elemental constitution of the worn zones. Specimen surface roughness and morphology of worn zones were evaluated by VEECO di-CPII atomic force microscope (AFM). All images were acquired in contact AFM mode using a symmetrically etched silicon-nitride probe. The scan size was 90 × 90 μm while scan rate and set point were kept at 0,5 Hz and 225 nN respectively.

The specific wear rate *K* was calculated for all regimes according to equation (1).

$$K = \frac{V}{F \cdot s}, \tag{1}$$

where *F* presents applied normal force, *s* sliding distance, and *V* worn volume which was calculated by equation (2).

$$V = L \cdot \left\{ r^2 \cdot \arcsin\left(\frac{x}{2r}\right) - \frac{x^2}{4 \cdot \tan\left[\arcsin\left(\frac{x}{2r}\right)\right]} \right\}, \tag{2}$$

where, *r* radius of alumina ball, and *x* worn channel width, as shown in Fig. 1b. Worn volume *V* was determined by measuring the wear tracks formed during reciprocating sliding and including the dimension of the alumina ball, Fig. 1a. Wear tracks generated by ball-on-plate tribo tests usually have non-uniform cross section along the tracks. Thus if worn volume calculation error is to be avoided a representative part of the wear track has to be analysed. In this study the middle of the wear track was considered as a representative since only in this region the real sliding speed is the one desired by the experiment. The representative worn volume was calculated for the wear track length of 90 μm, since all channels were imaged using 90 × 90 μm^2 scan sizes, Fig. 1a. Sliding distance s was calculated by multiplying the length of scanned area (*L*) with the number of sliding cycles.

3 Results and discussion

Average coating thickness of 1 μm was calculated for all studied specimens. The hardness of steel substrates was measured prior to the deposition process to a value of 740 HV. Therefore, material has high load bearing capacity and is appropriate for hard coating substrate. The coating hardness was measured to a value of 1560 $HV_{0,003}$ which is a satisfactory value, considering that the deposition is carried out at nearly room temperatures.

The surface topography of as-deposited coatings was acquired by the AFM and the following values of average roughness were obtained: Specimen 1 *Ra* = 53 nm; Specimen 2 *Ra* = 20,1 nm and Specimen 3 *Ra* = 3,5 nm. These values correspond to the one usually obtained by fine grinding or honing. The fine surface finish used in the present study was rarely applied in previous studies of tribological behaviour of TiN coatings during reciprocating sliding.

Fig. 2 shows the friction vs. the sliding time curves of TiN coatings tested at a load of 100 mN with a sliding speed of 15 mm/s. The friction factor showed similar behaviour for all specimens and for different regimes applied during the testing. For the tested range of conditions the beginning of the sliding was characterized by very low values of the friction factor (as low as 0,1).

All friction curves exhibit two stages, running-in stage and steady-state stage without evidence of a typical transition stage. In the running-in stage the friction factor smoothly increases with sliding distance. The smooth increase of friction factor is a desirable property for the practical application of TiN coatings. After deformation of asperities in contact a steady-state stage is reached where the friction factor is oscillating around constant value. The typical transition stage is absent, hence in the presented curves the transition stage is basically an extension of the running-in stage. By decreasing the coating roughness a sliding distance in the running-in stage increased, what means that the running-in stage prolonged. Therefore, for the smoothest specimen the friction factor maintains its growing tendency (Fig. 1c), even after reaching the maximum sliding distance used during the wear tests.

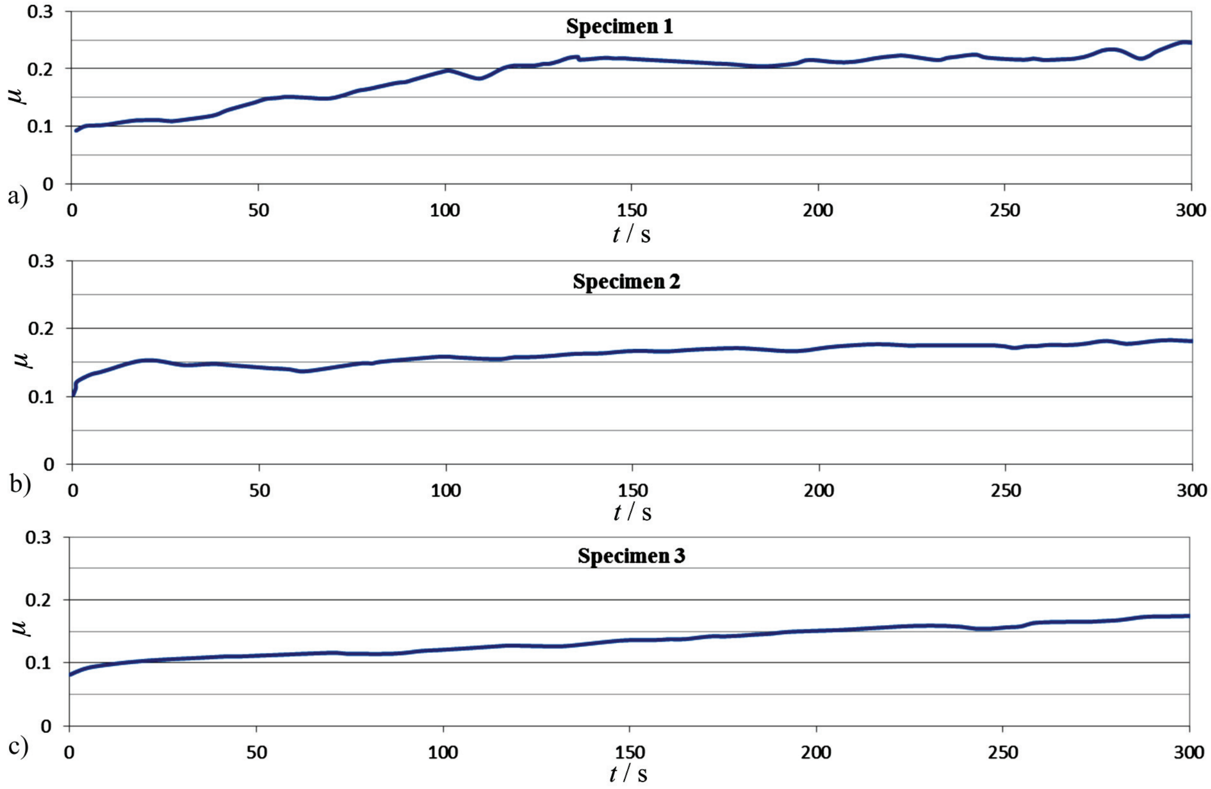

Figure 2 Evolution of the friction factor for different specimens, v = 15 mm/s, F = 100 mN

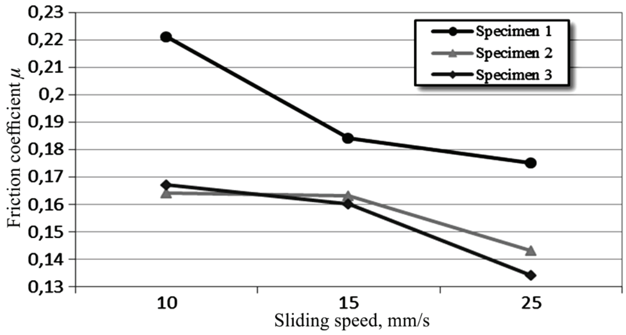

Figure 3 Friction factor of TiN coatings with different roughness in function of sliding speed

Fig. 3 summarizes the friction factor of TiN coatings with different roughness in the function of speed applied during the tribo-tests. The obtained low values of friction factor are typical for TiN coatings sliding against Al_2O_3 [8]. This can be explained by the presence of an oxide tribolayer on the coating surface during sliding which was revealed by EDX analysis [10]. Table 1 and Fig. 4 represent the typical result obtained by EDX analyses, conducted at the bottom of all worn zones. As there were no other elements present except Ti, these oxides are probably TiO_2. Formation of TiO_2 layer on TiN coating surface is common for sliding conditions used in the present study [15]. Temperature rise during reciprocating test with shorter stroke lengths favours tribo-chemical interaction between sliding pairs and surrounding atmosphere. The TiO_2 layer has low shear strength [11, 12] which leads to a low friction force [13, 14]. In addition EDX compositional analysis did not reveal the presence of Al in the worn zone. This indicates that the material transfer from the alumina ball did not occur, while the alumina ball is harder than TiN coating.

The friction factor decreases with increase in the sliding speed for all specimens (Fig. 3). Such results are contrary to findings of S. Y. Yoon et al. [9] who studied wear of TiN coatings. At higher sliding speed the tribolayer forms more easily [16]. It increases in thickness and keeps the friction factor low. The smoother surfaces give a lower friction factor, Fig. 3, but there was no significant difference in friction factor of Specimen 2 and Specimen 3. It appears that below a certain roughness, here *Ra* about 20 nm, the friction is less influenced by surface roughness.

During sliding contact, asperities of two bodies in contact interact with each other. This interaction can result in plastic deformation of asperities of one or both bodies. For rougher surfaces higher energy is required for asperity deformation resulting in higher friction.

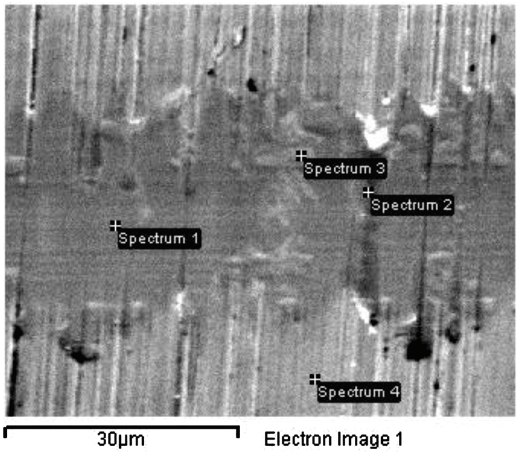

Figure 4 SEM micrograph of worn channel generated on Specimen 1, v = 15 mm/s, F = 100 mN

Table 1 Results of the EDX analysis carried out at the bottom of the worn channel at Specimen 1, related to Fig. 4

	Ti / %	N / %	O / %
Spectrum 1	74,30	16,69	10,01
Spectrum 2	63,98	11,90	24,13
Spectrum 3	65,32	12,29	22,39
Spectrum 4	76,55	18,22	5,23

Fig. 5 illustrates the morphology of worn zones formed on all tested specimens by sliding with different speeds at the load of 100 mN. The mild loading conditions applied did not produce any sign of hazardous wear. There were neither cracks, flakes nor fragmentation in the worn zones of studied specimens. This behaviour can be attributed to high coating toughness which was confirmed by qualitative indentation tests conducted in our previous research [17]. The maximum wear track depth was around 150 nm which is far less from the coating thickness of 1 µm. The wear debris was not present inside the worn zones. Detailed analysis of areas inside and outside the wear tracks revealed drop in surface roughness. The maximum depth (Rp_v) of the same machining ridges was lower inside the wear track. The machining ridges were flattened by plastic deformation and mild wear. The depth of worn channels was larger than the maximum depth of machining ridges. This suggests that machining ridges were pressed into the surface before being worn and plastically deformed. According to the SEM and AFM analyses there was no sign of wedge formation during wear. A wedge, which should be generated at the end of the wear track, forms when brittle coating is unable to absorb the load by its plastic deformation.

In addition to friction factor, wear loss is the other important parameter for practical application of hard coatings. The wear loss is often presented by worn volume. However, specific wear rate is more practical parameter for wear characterization. In order to calculate specific wear rates it is necessary to measure the worn channels. The accurate measurement of produced channels by optical or scanning electron microscopy is not an easy task. In order to overcome this problem atomic force microscopy has been applied in the present study. The worn zones produced during wear at different sliding speeds differ in size, morphology and roughness (Fig. 5). Increase in the sliding speed led to increase in width and depth of wear tracks. The dimensions of worn channels were measured by applying appropriate image processing software. Fig. 6 shows the measuring method applied. The conducted measurements provided the data for calculation of specific wear rates.

According to literature, a decrease of the wear rate with decrease in the friction factor should be expected [16]. Nevertheless, such relationship cannot be taken as a rule of thumb for all material combinations in sliding contact [18, 19]. Fig. 7 presents wear rates of coatings with different surface roughness in function of sliding speed applied during reciprocating sliding tests. The wear rate increases with increase in sliding speed and reaches a maximum of $72{,}3\times10^{-6}$ mm^3/N·m at the smoothest specimen tested with sliding speed of 25 mm/s. The obtained wear rate values are comparable to those usually observed for TiN coatings sliding against Al_2O_3 [8]. The two rougher specimens (Specimen 1 and 2) behaved similarly during sliding wear tests and exhibited almost the same values of specific wear rates. It appears that when sliding is conducted on surfaces of $Ra \approx 25 \div 50$ nm the surface roughness does not affect the coating wear behaviour significantly. In addition, for sliding speeds bellow 15 mm/s the wear rate is less affected by the surface roughness.

As already mentioned, the highest wear rate was calculated for the smoothest specimen. This value was significantly higher than the wear rate values of other samples. Although the wear tracks formed on the smoothest sample tested with speed of 25 mm/s were the widest, they were also of the most irregular shape (Fig. 4). The more the profile of the wear track deviates from the arc-like shape, the greater error is incorporated in the calculation of the specific wear rate. Therefore, it is to believe that the actual wear rate of the Specimen 3 tested with 25 mm/s should be at least half of the calculated one. Since there is no great difference in the wear rate of the two rougher specimens, the high wear rate value determined for the smoothest specimen could be considered as the experimental scatter. However, the sliding tests were conducted two times, and both times the same results were obtained.

This result is contrary to the usually observed increase of the wear rate with increase in the surface roughness [22]. Higher stresses, which are present on rougher surfaces, can lead to formation of cracks and flakes, coating fragmentation, formation of fatigue pits, and as a result to more severe wear. Neither of these defects was observed in the present study. The higher wear rate on the smoothest specimen can be explained in the following manner. During the test with the same applied load and the same sliding speed the same amount of energy is dissipated. On the rougher specimens the large amount of the energy is used for deformation of the machining ridges, while on the smoothest specimen almost all energy is used for formation of micro grooves present in the wear track of this specimen (see Fig. 5). The difference in the wear rates was a consequence of different wear mechanisms acting on the tested specimens. While ridge deformation and mild abrasion were acting on the rougher specimens, abrasion was the dominant wear mechanism on the smoothest specimen.

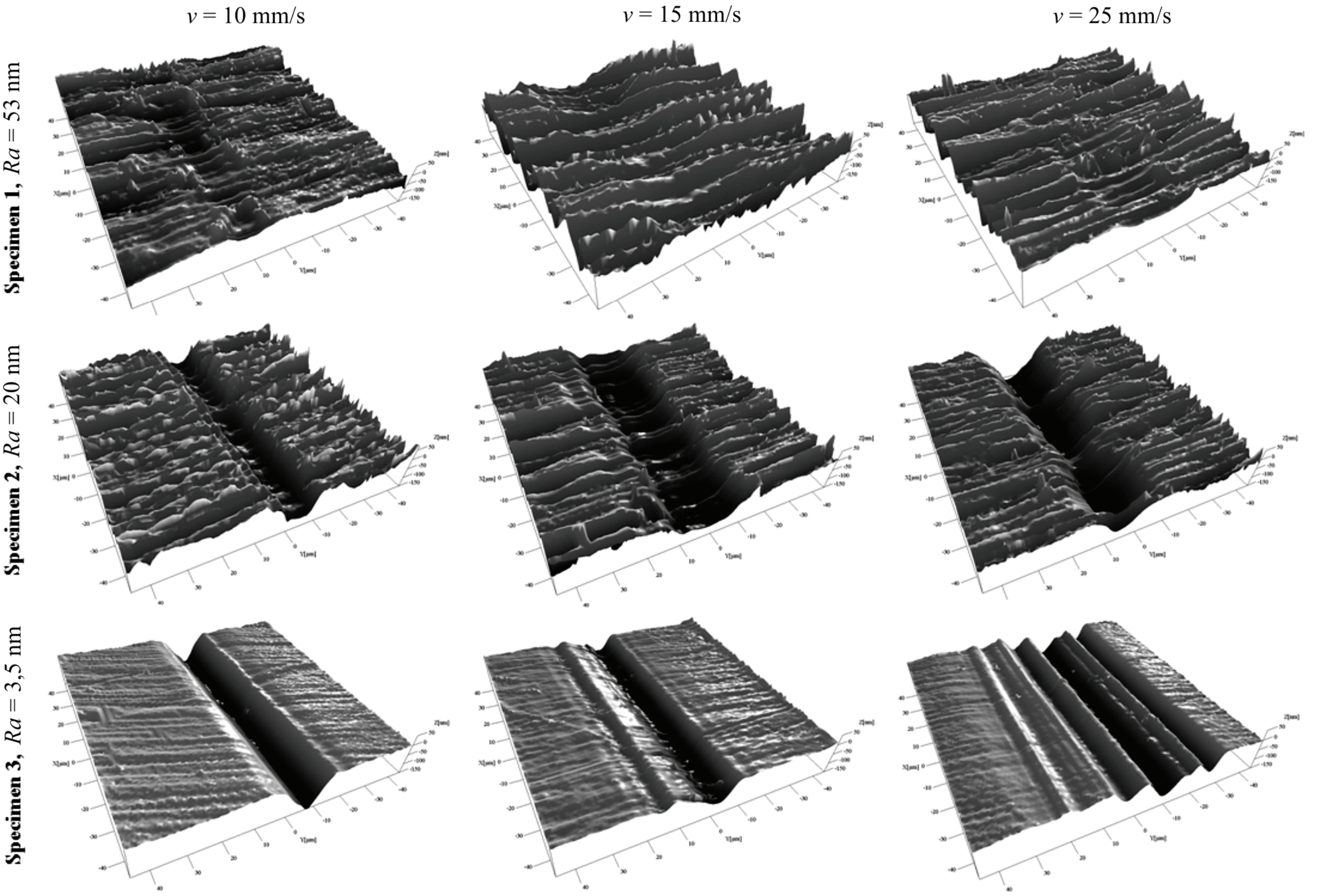

Figure 5 Morphology of the worn zones generated on TiN coatings of different roughness by reciprocating sliding with different sliding speeds

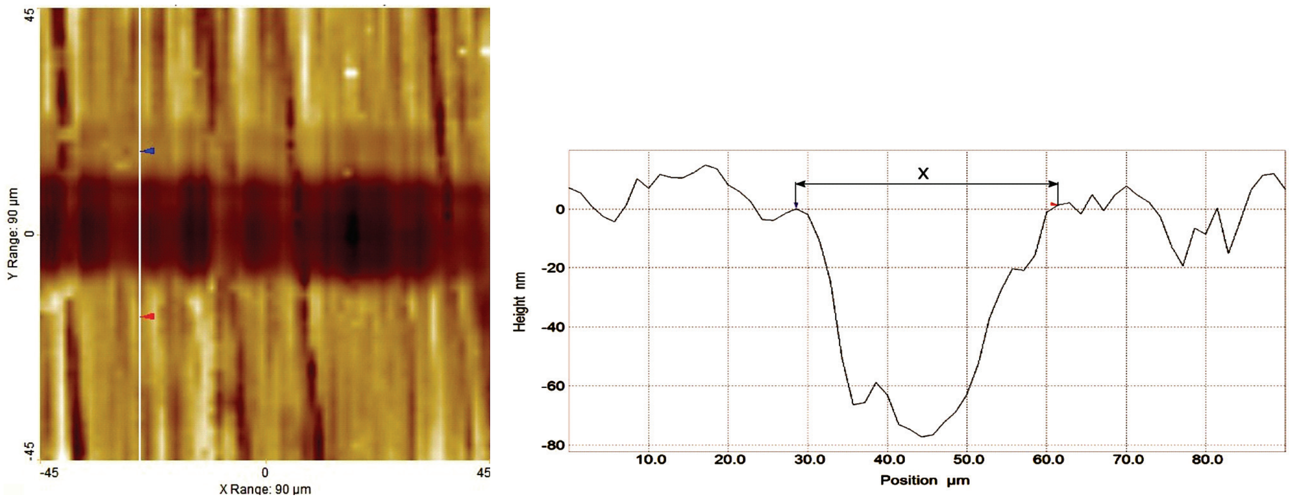

Figure 6 Measurement of wear track width

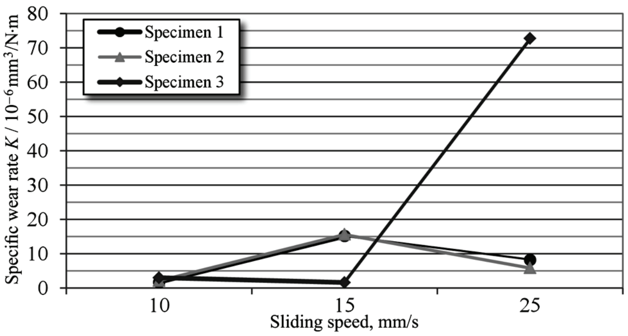

Figure 7 Specific wear rate of TiN coatings with different roughness in function of sliding speed

According to the results of this investigation, for the conditions applied, Specimen 2 has the optimum surface finish. This specimen exhibited low friction factor and the lowest wear rate. Although the polished specimen exhibits the lowest friction factor, the costs of its machining are not justified in terms of high wear rate it displays in sliding wear. These findings are of practical importance for application of TiN coating as a wear resistant coating.

The roughest specimen was submitted to further examination of the relation between sliding direction and machining ridges. There was no significant difference in the friction factor when sliding transversal (0,221) and parallel (0,222) to the ridges. Such behaviour is preferred in industrial applications as the relation between loading direction and machining ridges does not have to be considered during the designing stage of a particular part.

4 Conclusions

Tribological behavior of the TiN coatings of different roughness has been investigated on a reciprocating sliding against Al_2O_3 ball. The effect of sliding speed on the wear behaviour of the coatings is discussed. The following conclusion can be drawn based on the experimental results:

- Low values of friction factor were obtained, ranging from 0,13 to 0,24. Low values of the friction factor are attributed to the presence of titanium oxide inside the wear tracks.
- The friction factor of coatings with different roughness decreases with increase in sliding speed. The roughest specimen exhibited the maximal friction coefficient. For *Ra* below 20 nm the friction factor is less affected by surface roughness.
- Increase of wear rate with increase in sliding speed was observed. The highest value of wear rate was calculated for the smoothest specimen. There was no significant difference in wear behavior of specimens with average roughness between 20 and 50 nm. For this range of surface roughness, the wear rate is less affected by sliding speed applied during the tests.
- There was no sign of crack and flake formation, fragmentation or fatigue pit formation on any of the tested specimens. The wear mechanism of the TiN coatings deposited on rough substrates was plastic deformation of machining ridges combined with mild abrasion, while abrasion was the dominant wear mechanism on the smoothest specimen.
- The specimen with average surface roughness of 20 nm has exhibited the best tribological behaviours. The low friction factor was observed and the lowest wear rate was calculated for this specimen.
- The change of sliding direction relative to the direction of machining ridges does not influence the wear behaviours significantly.

In order to construct wear maps, future research will be oriented to studying the coatings deposited on substrates with wider range of surface roughness in tests with wider range of sliding speeds.

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