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Tribological performance of TiN and TiCN coatings on a working tool steel †

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Abstract

Although the applications of TiN and TiCN coatings are extensive, their mechanical and tribological properties are influenced by the substrates in which they are deposited. The present work is focused on the tribological performance of TiN and TiCN coatings on a working tool steel. Besides, adhesion and microhardness tests were carried out. The adhesion performance of both coatings resulted in class 1, according to CEN/TS 1071-8 standard, which allows observing the quality of adhesion. The composite microhardness was investigated by the analysis of relative indentation (β). Pin-on-disk tests were performed in dry and lubricated condition at 100 °C against tungsten carbide (WC). Low friction coefficients of μ_k = 0.08 for TiN and μ_k = 0.03 for TiCN were obtained in lubricated conditions. Wear mechanisms were analyzed by scanning electron microscopy (SEM). Abrasive wear was observed as the principal wear mechanism in dry condition, while in lubricated conditions wear signals seem to be scarcely noticeable.

Keywords: Adhesion; Microhardness; TiCN; TiN; Tribology

1. Introduction

Hard coatings remain in focus due to their versatility characteristic in many different research areas [1-3]. Coating materials such as titanium nitride (TiN) and titanium carbonitride (TiCN) have gained much attention due to their mechanical compatibility with various metallic substrates. However, the preparation of these ceramics on the metallic substrates presents challenges due to the differences in mechanical properties mainly in tribological and mechanical performance [1-6]. Therefore, significant investigations have been accomplished with the goal to improve the properties of these coatings to broaden the applications of them. TiCN coatings have high thermal stability, excellent mechanical and wear behavior. Besides, they have shown low internal stress and high corrosion resistance [4, 6, 7]. Moreover, TiN coating is considered one of the most applied hard ceramic coatings for different tribological applications [5, 8-10]. Currently, both TiN and TiCN coatings continue to be used for protection of mechanical elements and cutting tools materials [8-11].

On the other hand, AISI H13 steel is a well-known hot-working tool steel commonly used to make hot extrusion dies, extrusion mandrels, hot work punches and forging tools due to its excellent mechanical properties. Despite them, due to the extreme working conditions such as high stress, temperature and resilience time, the service lifetime performance of H13 tools is affected by some failure mechanisms. Thermal fatigue, abrasive, and adhesive wear on the surface of the working tools are the main results of this working conditions. Thus, the working tools are inevitably replaced after a period, affecting the total cost of the manufacturing processes [12, 13]. Therefore, several studies have been carried out to improve the wear resistance of the AISI H13 steel surface by nitrocarburizing methods, cryogenic treatments, and laser beam irradiation [12-14].

The goal of the present work is to investigate the behavior of TiN and TiCN coatings on AISI H13 substrate, to extend the applications of these protective coatings in the manufacturing processes. For that purpose, microhardness and adhesion tests have been performed, the tribological behavior in lubricated condition with temperature was also investigated, and it was compared in dry condition.

2. Experimental procedure

2.1 TiN and TiCN coatings deposition

For the present study, AISI H13 steel (0.4 wt.% C, 4.9 wt.%

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Cr, 0.5 wt.% Mn, 1.25 wt.% Mo, 1 wt.% Si, 0.9 wt.% V and Fe balance) was used as substrate. The steel was machined into small cylinders of 25 and 5 mm in diameter and thickness, respectively. The substrate samples were hardened and tempered reaching a hardness value of 52 HRC (5.33 GPa). Then, these substrates were polished in a wet grinding process by using different grinding papers to obtain a surface roughness of 0.09 μm in Ra. For that purpose, the samples were rotated 90º before using the following grinding paper, to remove the grinding marks above. Subsequently, by contact profilometry, it was determined the average roughness of the specimens. Twenty measurements were performed to obtain an average roughness of the sample surface by using a surface roughness tester. Posterior to the roughness preparation, the samples were coated with TiN and TiCN by physical vapor deposition PVD with cathodic arc by using a system bias and cathodic arc evaporation. The coatings were applied by a specialist coatings supplier, obtaining a layer of 5 and 2.5 μm of thickness for the TiN and TiCN coating, respectively. The temperature was 480 °C for the two coatings processes. The selected thicknesses are very close to those found in the Refs. [9, 10, 15, 16]. On the other hand, TiCN is well known to exhibit a combination of the best properties of TiN with those of TiC [4-7]. For that reason, in this work, the thickness of the TiN coating was doubled in order to improve the TiN performance in friction and wear conditions.

The element concentration of TiN and TiCN coatings were determined by using a scanning electron microscope (SEM) coupled with energy dispersive X-ray spectroscopy (EDS). Fig. 1 shows EDS analysis with the element concentration corresponding to each coating. The thickness of each coating was also corroborated by SEM image analysis from the cross-section of the TiN and TiCN coatings, respectively (Fig. 2). The thickness of 5 μm for TiN and 2.5 μm for TiCN coatings were measured. It can be observed that the first one exhibits a more homogeneous thickness than TiCN coating. The surface roughness after the coating deposition was 0.25 μm in Ra for both coatings.

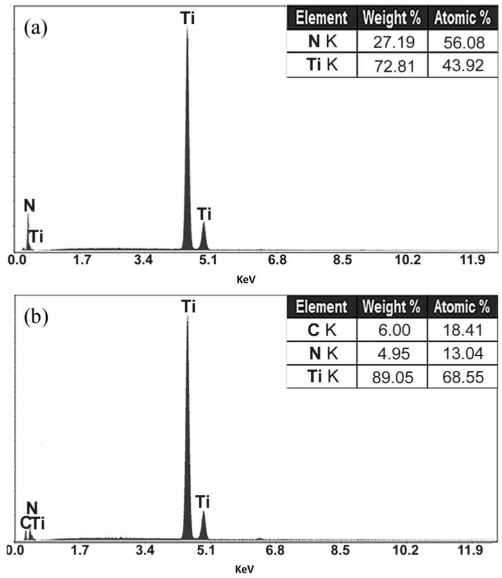

Fig. 1. EDS analysis and element concentration of (a) TiN; (b) TiCN coatings on AISI H13 steel.

2.2 Microhardness and adhesion measurements of TiN and TiCN coatings on AISI H13 steel

Microhardness measurements were performed by Vickers indentations using a Microindenter Vicker-Knoop with a load range from 0.98 to 9.8 N for 15 seconds. Five measurements were taken at each load. The imprints of each test were analyzed by using an optical microscope. The lengths of the diagonals were measured. Then, the microhardness value HV was calculated from the average diagonal length d and the load P of the indentations, from:

$$HV = \frac{(1.8544)P}{d^2} . \tag{1}$$

The influence of the substrate hardness on the film hardness was studied by the investigation proposed by Tuck et al. [17]. There, it was determined the dimensionless parameter beta (β). Which corresponds to the ratio between δ and the thickness coating t, where the indentation depth, δ, is given by the ratio d/7. Adhesion quality between AISI H13 substrate and coatings was evaluated by using Rockwell indentation standard test CEN/TS 1071-8 [18]. Five indentations were performed using a load of 100 kg_f for each coating. The indentation imprints were observed by SEM images, and then, they were related to the corresponding classification according to the standard.

2.3 Tribological investigation

The tribological performance was investigated by friction tests by using a tribometer with a pin-on-disk configuration. In this configuration, the pressure was applied on the rotating disc through a static ball-shaped pin. The experiments were performed in dry condition at room temperature, maintaining 30 % of humidity. On the other hand, for lubricated conditions, it was used a mineral lubricant with graphite nanoparticles at 100 ºC (this lubricant is used in the die and forging process and is commercially available). Commercial tungsten carbide (WC) pins of 6 mm in diameter were used as counterparts. The WC pins have an elastic modulus of 670 GPa and a microhardness value of 1370 HV. The results of friction and wear tests were the average of five readings to consider the repeatability. The normal load applied was 10 N; it corresponds to a contact pressure between the substrate and WC pin (1790 MPa) near to the yield stress of AISI H13 steel. The sliding distance, sliding speed, and wear track radius were established at 1000 m, 0.025 ms^{-1} and 2 mm, respectively. The kinetic friction coefficients (μ_k) were registered continuously

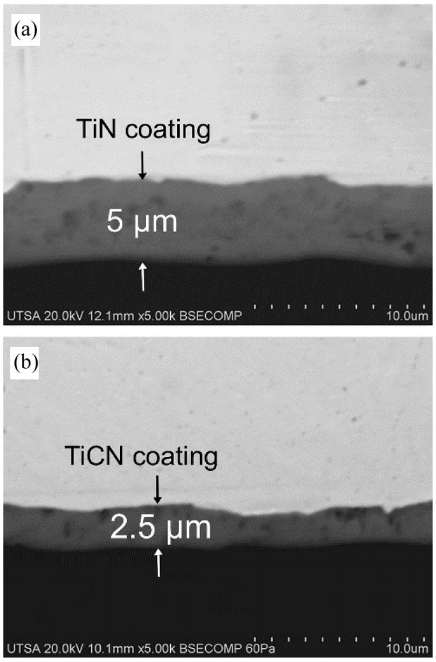

Fig. 2. SEM images of cross section of (a) TiN; (b) TiCN coatings on AISI H13 steel.

during the entire test by Tribox 4.1 software. Wear rate of samples was calculated according to:

$$K = \frac{2\pi r A}{FS} \qquad (2)$$

where A is the cross-sectional area of wear track which was determined by contact profilometry, r is the wear track radius, F represents the applied normal load and S corresponds to the total sliding distance. Finally, wear mechanisms were also investigated by SEM technique.

3. Results and discussion

3.1 Adhesion and microhardness tests

A comparison of imprints produced by Rockwell indentation tests on the TiN and TiCN coatings is shown in Fig. 3. For TiN coating (Figs. 3(a) and (b)), it can be seen many small radial cracks (50 μm) around the imprint. Unlike the TiN coating, longer radial cracks about 150 μm in length were observed on TiCN coating (Figs. 3(c) and (d)). This behavior was expected since the hardness of TiCN coating is higher than that of TiN coating, making it less deformable and therefore more fragile and susceptible to crack. Besides, it could be related to the thickness coating. As the TiCN coating is 50 % thinner than TiN coating, it could impact the formation of large cracks observed on TiCN coating. Furthermore, it has been reported that the mechanical properties of the substrate affect the performance of coated systems [19]. In fact, a way to limit this phenomenon is reducing the difference in hardness between substrate and coating. Besides, it has been

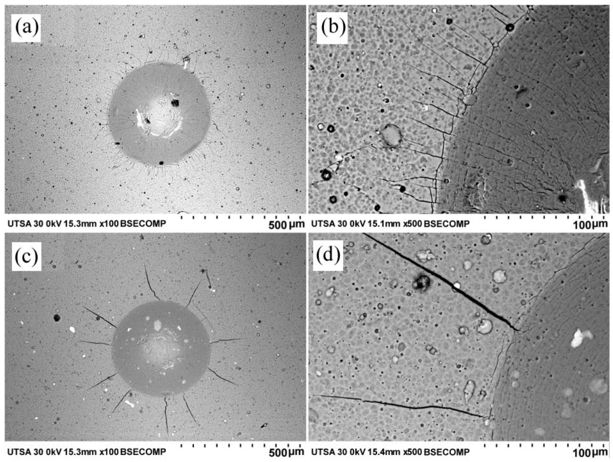

Fig. 3. Imprints of Rockwell indentation tests for evaluation of adhesion of (a) and (b) TiN; (c) and (d) TiCN coatings on H13 tool steel substrate.

investigated that if the surface to be coated has high plasticity and low toughness; the coating would be sagged and collapsed under high specific loads during the friction performance [18]. Although cracks in both coatings are present, no coating failure was observed. The relationship between the imprints of the indentations and the classification given by the standard [18] represents an adhesion Class 1. This classification points out a well-adhered coating due to the high interfacial bonds on substrate/coating pair.

Fig. 4 displays the hardness as a function of the applied load, showing the variations of hardness values for TiN and TiCN coatings on H13 steel. It can be seen higher values of hardness for TiCN than for TiN coating which are in the range of 14.9 to 12.2 GPa and 11.3 to 8 GPa for TiCN and TiN coating, respectively. A gradual diminution in hardness can be observed in both coatings with the increase of load indentation. There, the hardness of the substrate influences the hardness of the coating. In this point, it is convenient to define the β parameter to identify that contribution [17]. Values of $\beta > 1$, imply that the indenter deformed and penetrated the coating; thus, the substrate properties dominate the system response. When $\beta < 0.1$ the influence of the substrate on the deformation is small; and for this and lower indentation depths, only the response of the coating is observed. Therefore, values of β in the range between 0.1 and 1, are of most interest to study the hardness substrate contribution [17]. In that sense, the inset in Fig. 4 plots the ratio β vs. the applied load for both TiN and TiCN coated H13 steel substrates. This figure shows few values in this range at low loads of indentation. Above 3 N there is a higher substrate contribution for these applied loads, at this point, the microhardness measurements at the lowest load would be only of the coatings contribution. It has been reported a wide range of microhardness values for TiN and TiCN. These values can vary depending on the fabrication conditions for both coatings, as well as the carbon concentration for TiCN coating. Although that, our results (1153 $HV_{0.1}$

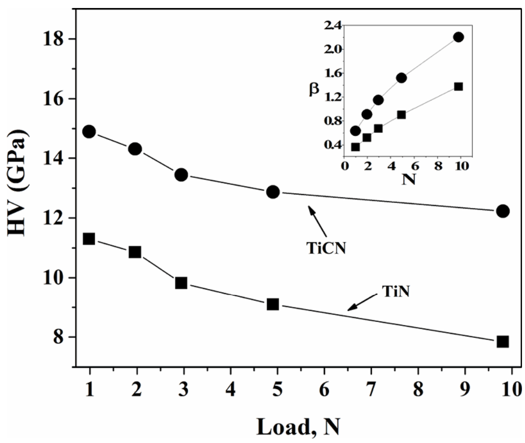

Fig. 4. Hardness and β parameter vs. applied load of TiN and TiCN coatings.

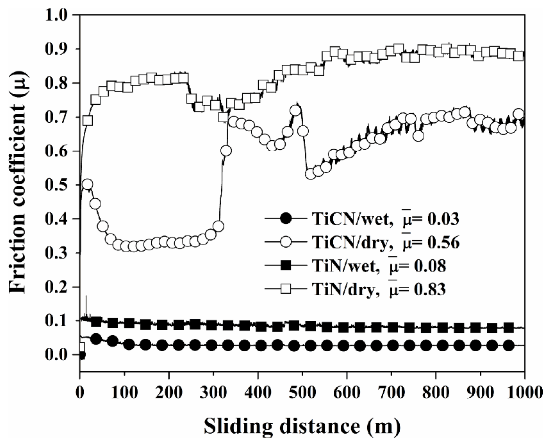

Fig. 5. Friction coefficients of TiN and TiCN coatings in dry sliding contact at room temperature and wet condition at 100 °C.

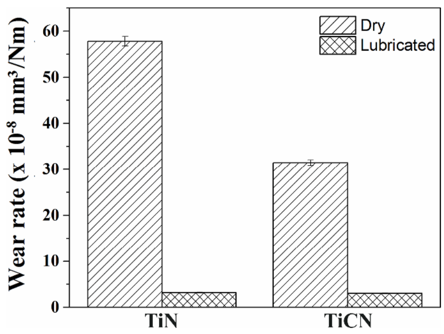

Fig. 6. Wear rates of TiN and TiCN coatings.

for TiN and 1520 $HV_{0.1}$ for TiCN) are close to those reported recently by Mi et al. [20]. They obtained microhardness values of 1311 $HV_{0.1}$ and 1674 $HV_{0.1}$ for TiN and TiCN coatings, respectively; with thicknesses higher than the coatings of this work.

3.2 Friction and wear behavior of TiN and TiCN coatings in dry and lubricated conditions

The friction coefficient variation of TiN and TiCN coatings in dry and lubricated conditions is shown in Fig. 5. In dry friction, it can be seen an unstable behavior on TiN and TiCN coatings during the first stages of the tests. At the first stage (to 250 m) both coatings exhibit lower variation, showing friction coefficients values around $\mu_k = 0.83$ for TiN and $\mu_k = 0.32$ for TiCN. Above 300 m, the friction coefficient of TiN coating increases gradually with the sliding distance, reaching the steady stage after 700 m; with a coefficient value of $\mu_k = 0.89$. Meanwhile, TiCN coefficient exhibits an increase up to 500 m. Above this phase, there is a gradual increase with the distance reaching an ending value of $\mu_k = 0.7$. This unstable behavior, for both coatings, could be associated with the presence of particles in the coating surface due to the detachment of material. Also, can be appreciated lower friction coefficients for TiCN than TiN, this behavior is no stranger. Several works have been reported lower friction coefficient for TiCN than TiN under different tribological conditions [9-11, 19]. This effect could be related to the fact that the presence of carbon in the TiCN coating works as a solid lubricant reducing the friction [10, 20]. On the other hand, in the lubricated condition, the friction coefficients of both coatings decreased considerably ($\mu_k = 0.08$ for TiN and $\mu_k = 0.03$ for TiCN). Although the friction coefficients are relatively close between them, in comparing with the friction values observed in dry condition, the friction coefficient of TiCN coating remains lower than that of TiN. This effect has been seen by other researchers who have reported low values of friction coefficient in lubricated media such as water, artificial seawater, and synthetic lubricants for both TiN and TiCN coatings [10, 15, 16]. However, the friction coefficients reported by them are much higher than those reported in this work, i.e., Shan et al. [10] reported friction values $\mu_k \approx 0.10$ for both coatings. Meanwhile, Lorenzo-Martin et al. [16] evaluated the friction and wear performance for TiCN and TiN on carburized and hardened 4120 steel using different formulated lubricants. They also obtained higher friction coefficients with variation at the initial stages, reaching a steady state after 30-60 min of the test duration. The friction coefficients that they reported were ranging approximately from $\mu_k = 0.15$ to $\mu_k = 0.08$ for TiCN coating, and from $\mu_k = 0.17$ to $\mu_k = 0.065$ for TiN. They have used a synthetic transmission lubricant that has an additive for wear protection. The above differences in friction coefficient values could be attributed to the lubricant properties. Taking into account that the fluid film supports a part of the normal load, and part of the friction force is backed up by the friction force viscous of lubricant film [21]. Moreover, the conditions of friction tests have also an influence on the measurement parameters; for example, the above works mentioned were performed with a lot of lesser sliding time and conducted in reciprocating sliding contact.

Fig. 6 represents the wear rates values for TiN and TiCN coatings, showing agreement with the friction behavior in dry conditions. The values corresponding to TiN coatings are

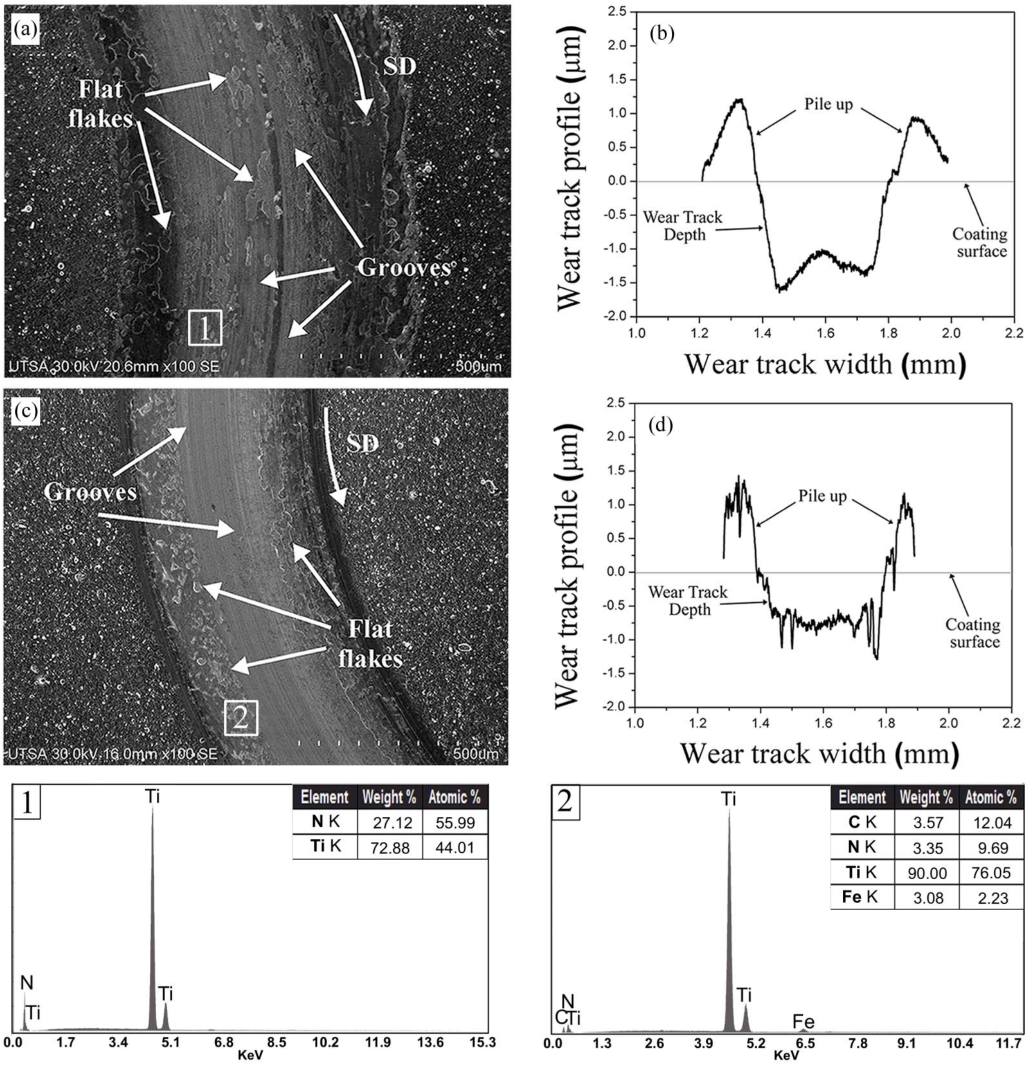

Fig. 7. SEM micrographs, wear track profiles and EDS (corresponding to number 1 and 2) of worn surfaces for (a) and (b) TiN; (c) and (d) TiCN coatings in dry sliding contact.

around of 60x10^{-8} mm^3/Nm, while for TiCN coatings are 30x10^{-8} mm^3/Nm, approximately. Whereas for lubricated conditions, the wear rate values decreased considerably until they reached 3.18x10^{-8} mm^3/Nm and 3.02x10^{-8} mm^3/Nm for TiN and TiCN coatings, respectively. These values are lower than those reported recently by other works [10].

Fig. 7 exhibits the wear scars for TiN and TiCN coatings in dry conditions. The SEM micrographs show the wear track surfaces while the wear track profiles show widths around 0.42 mm for both, TiN and TiCN coatings (Figs. 7(b) and (d)). Besides, it can be observed that the wear track depths are larger for the TiN coating than for the TiCN one. This difference of 0.5 μm in depth involves larger wear rates for TiN coatings of approximately twice the TiCN values, as it was observed in Fig. 6. The lower depth on wear tracks of TiCN coatings can be explained by the study of Leyland and Matthews [22]. They established that the H/E ratio is a reliable indicator of good resistance in a coating. The H^3/E^2 ratio is proportional to a resistance of the material to plastic deformation during contact, and higher H^3/E^2 ratio values mean better resistance to mechanical failure [23]. Therefore, at the first instance, the H^3/E^2 ratio was determined using the hardness values of 24 GPa for TiN and 30 GPa for TiCN. These values correspond to the hardness of the coatings only. Values for this ratio of 0.18 and 0.21 were obtained for the TiN and TiCN coatings, respectively. However, in a real application, where a coated component works as a substrate-coating system, it should be considered the hardness of such system. Whereby this ratio was determined again, but now with the hardness of the coated system (values of 11.3 GPa for TiN and 14.9 GPa for TiCN, from Fig. 4). With that consideration, ratio values of 0.023 and 0.027 had been obtained for TiN and TiCN coatings, respectively. Although these H^3/E^2 ratio values are out of the established by Tsui et al. [23], the value for the TiCN coating remains higher than that of TiN coating. This effect can also be seen in the wear mechanisms, which

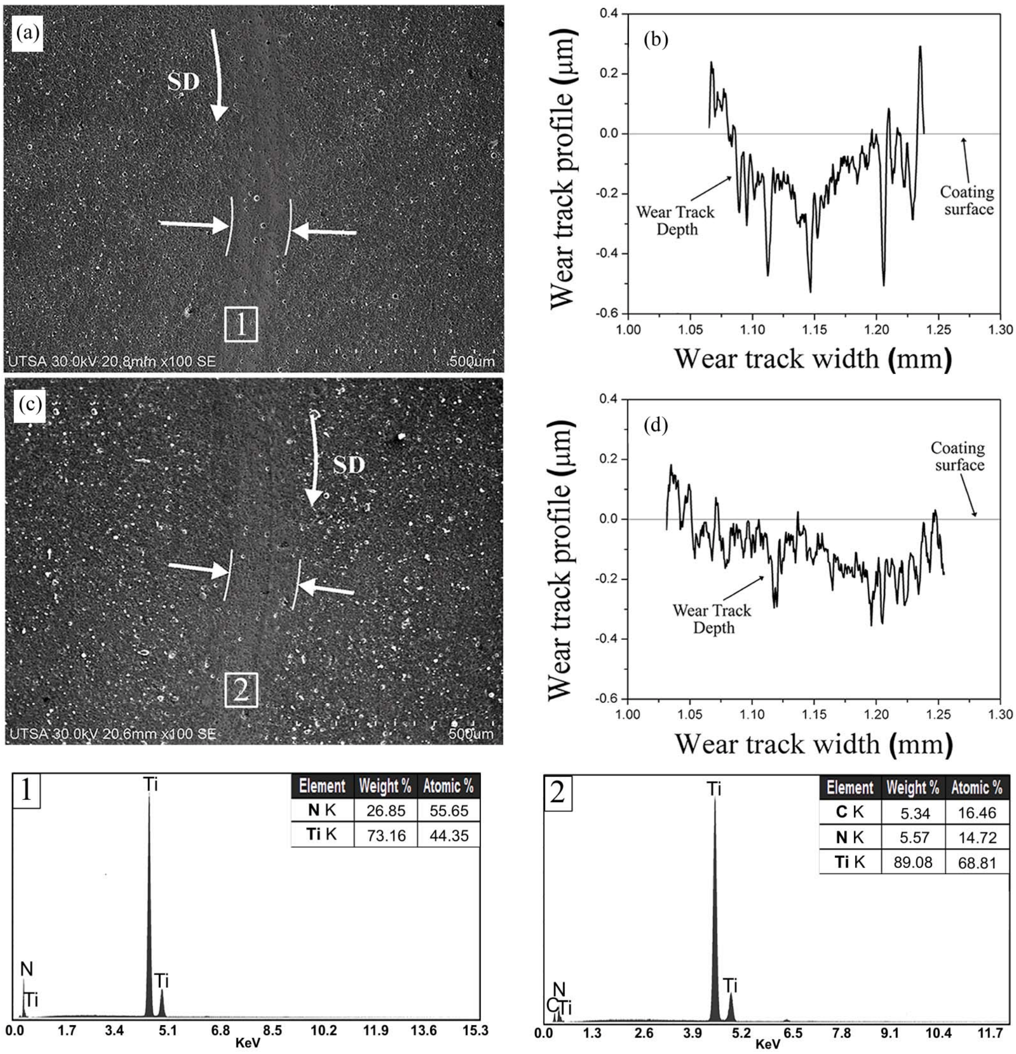

Fig. 8. SEM micrographs, wear track profiles and EDS (corresponding to number 1 and 2) of worn surfaces for (a) and (b) TiN; (c) and (d) TiCN coatings in lubricated condition.

are exhibited in Figs. 7(a) and (c), corroborating the better resistance of TiCN coating. These images show the worn surfaces micrographs in which the abrasive wear can be observed through the grooves parallel to the direction of sliding (plowing). That could be caused by particles trapped between the contacting surfaces or asperities of the counterpart. Also, the wear profiles of the worn scars for both coatings (Figs. 7(b) and (d)), show accumulation of material (pile up) on the edges of the track. Thus, as higher plastic deformation, higher pile up could be made. In that point, by measuring the area of the pile up regions, we obtained area values of 229.32 μm^2 and 155.55 μm^2 for the TiN and TiCN coating, respectively. It can be observed that the amount of plastic deformation suffered by the TiCN coating is 32.2 % less than that of the TiN coating which has a lower H^3/E^2 ratio value. Furthermore, some wear particles were removed from the contact area. Many of them were accumulated in the contact zone between the part and the counterpart of tribosystem, compacting and adhering inside the wear track as flat flakes. The EDS spectra shown in Fig. 7 correspond to number 1 and 2 indicated in the worn surfaces for TiN and TiCN coating (Figs. 7(a) and (c)). The EDS analysis presents small amounts of Ti and N, suggesting the presence of both coatings on the H13 substrate. Fig. 8 also depicts the wear scars, the wear track profiles and EDS analysis for TiN and TiCN coatings in lubricated conditions. In this case, as expected, the wear track widths as well as the depths of wear tracks, decreased considerably compared to dry condition because of the use of a lubricating medium. Unlike the wear tracks in dry conditions, there are no visible morphological changes on wear tracks in lubricated conditions. In Figs. 8(a) and (c) just light wear marks can be perceived which are point out between white arrows. Some parallel grooves could be present in the wear tracks and some plastic deformation as suggest the analysis of the profile pattern of the wear track (Figs. 8(b) and (d)). These grooves would indicate that the main wear mechanism was abrasion. However, since the EDS analysis of both coatings on the worn tracks (points 1 and 2 in Fig. 8), show only the element concentration corresponding to

TiN and TiCN coatings, respectively; this wear mechanism not affected the integrity of the coating.

On the other hand, durability is an important factor of a coating for the protection of the elements; the ideal coating must remain on the surface during more operation cycles. In this study, the percentage of coating that was worn during the cycles tested was calculated by the average depth of the wear scars (Figs. 7 and 8). The TiN coating was the less worn, 22 % without lubrication and 3 % in lubricated condition. Whereas, the TiCN coating was worn approximately 26 % and 5 % in dry and lubricated conditions, respectively.

4. Conclusions

Within this study, it has been investigated the effect of TiN and TiCN coatings on the tribological performance of AISI H13 substrate used as working tool in manufacturing processes. The results were as follows:

- The TiN and TiCN coatings showed well adhesion quality to the H13 steel substrate, which is essential for tribological performance. In the load range of 1 to 3 N in the hardness tests, a lower contribution of the substrate was shown in the hardness of both coatings.
- On the other hand, the coefficients of friction obtained, both, in dry and lubricated conditions were lower than those reported by other researchers for the same type of coatings with different conditions. Similarly, the wear factors calculated were minimal, in the order of 10^{-8}.
- Meanwhile, the primary wear mechanism was abrasion for both coatings. Also, some plastic deformation was shown in the wear track profiles.
- Under lubricated conditions, the integrity of the coatings was maintained as confirmed by SEM investigation and EDS analysis.
- The H^3/E^2 ratio supports the resistance to plastic deformation for TiCN during the sliding contact showing higher ratio than TiN.

In general, AISI H13 steel showed better friction and wear behavior with the TiCN coating than with TiN, despite having a 50 % lower thickness. The friction was reduced to 62.5 % and the wear to 50 % for TiCN coating. Besides, the values of the percentage of worn coating in both, dry and wet conditions, of TiCN are very similar to the TiN ones. Then, on the basis of these results, the TiCN coating is the most suitable coating for AISI H13 tool steel.

Acknowledgments

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Nomenclature

HV : Vickers microhardness
β : Relative indentation depth
δ : Indentation depth
t : Thickness coating
$\underline{\mu_k}$: Kinetic friction coefficient
μ_k : Average kinetic friction coefficient
K : Wear rate

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