J MATER RES TECHNOL. 2019;8(1):259–266

Available online at www.sciencedirect.com

jmr&t
Journal of Materials Research and Technology
www.jmrt.com.br

Original Article

Fretting wear resistance of DLC hard coatings deposited on nitrided martensitic stainless steel

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ARTICLE INFO

Article history:
Received 30 May 2017
Accepted 28 December 2017
Available online 24 March 2018

Keywords:
DLC hard coatings
Fretting wear
Adhesion

ABSTRACT

In this work, the fretting wear behavior and adhesion of DLC coatings deposited by PACVD on nitrided and non-nitrided martensitic stainless steels were studied. Fretting wear tests were carried out with different duration and loads and pin-on-disk was also performed. The adhesion was evaluated by Rockwell C indentation and scratch test with constant and variable loads. The coating thickness was 2.5 μm and the nitrided layer, 11 μm. The coating hardness and Young's Modulus were about 16 and 110 GPa, respectively. The duplex samples resulted with better adhesion than the only coated samples in all test conditions. In the scratch test, the critical load in the duplex sample was 58 N and in the coated sample, 12 N, showing a brittle failure mode. In the Rockwell C indentation test, the adhesion was better in the duplex sample because the nitriding treatment changed the failure mode of the system. The wear resistance was also related to adhesion, since in the experiments with high loads and long durations adhesive failures could be detected when stresses reached the interface and the substrate. In the duplex sample, the nitrided case, with higher load bearing capacity than the substrate, resulted in a better wear behavior of the system.

1. Introduction

AISI 420 stainless steels have different applications such as dental and surgical instruments, hydraulic components, vapor conduction, automotive components where good wear resistance and strength are required [1,2]. In the different applications where these steels are used, the fretting wear is one of the main mechanisms responsible for its degradation, therefore, it is important to improve and study resistance to this type of wear. Fretting is a contact degradation process that occurs due to reciprocal relative displacement between surfaces of two contacting components such as hubs and disks pressed-fitted to rotating shafts. The amplitude of the motion is in a range from 1 to 100 μm, and this phenomenon can cause

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https://doi.org/10.1016/j.jmrt.2017.12.004

failures as a result of wear damage, which is generated by the relative oscillation motions and cyclic stress loading [3–5].

Different treatments like coatings or surface modification processes can be performed in order to improve mechanical properties of steels. Diamond like carbon (DLC) coatings have low friction coefficient, good wear resistance and chemical inertness [6,7]. Wear behavior of DLC coatings deposited on steels or metallic substrates has been studied in different sliding situations but not much in fretting [7–10]. Differences in contact conditions, stress magnitude and displacement amplitude during fretting wear can produce different kinds of damage. As there are also many coatings properties and tests conditions that have influence on the fretting wear resistance, each coating and each test requires specific analysis.

On the other hand, the DLC coatings have adhesion problems when they are deposited on metallic substrates because carbon diffuses into the metals delaying the DLC nucleation. Moreover, the thermal expansion coefficients of coating and steel are not compatible, which causes poor adhesion [11]. Different interlayers or multilayers between the substrate and the DLC film have been proposed in order to improve the adhesion [12–14]. In addition, diffusion treatments such as plasma nitriding can be used as a previous treatment. Plasma nitriding allows not only surface hardening but also the modification of the composition profile, improving the adhesion as a consequence [11,15,16]. The duplex system (nitrided layer + DLC coating) can be then considered as a good option not only to enhance adhesion but also the fretting wear resistance [17–19].

As fretting wear can lead to loosening of joints, resulting in increased vibration and consequent acceleration of damage in contact components, the durability of a coating should be assured. The application of DLC on stainless steels is growing but some issues about adhesion and wear resistance of duplex coatings remain unclear. In this work, fretting wear resistance with different test duration and loads, and adhesion tests were carried out on DLC coatings deposited on nitrided and non-nitrided martensitic stainless steel, with the goal of adding some scientific and technological knowledge to this field.

2. Experimental

AISI 420 martensitic stainless steel, with a chemical composition of 0.38 wt% C, 13 wt% Cr, 0.44 wt% Mn, 0.42 wt% Si, 0.07 wt% Mo, 0.02 wt% P and Fe as balance, was used as base material. The samples were of 2 mm thick and 25 mm diameter disks, cut from an AISI 420 martensitic stainless steel plate. The samples were heated up to 1030 °C, quenched in agitated air and double tempered at 260 °C for 2 h according to the standards [20]. The nitriding process was performed in an industrial equipment at IONAR S.A. (Argentina) using a DC pulsed discharge for 10 h at a temperature of 390 °C using a gas mixture composed of 20% N_2 and 80% H_2. The DLC films were deposited by PACVD process (Plasma Assisted Chemical Vapor Deposition) at INPE, Brazil, with an asymmetrical bipolar DC pulsed discharge, using methane as the precursor gas at 150 °C for 2 h. Previously, a thin amorphous silicon interlayer was deposited using silane gas as the precursor.

The coating was characterized by Raman spectroscopy, optical microscopy and SEM. The hardness (*H*) and Young's Modulus (*E*) of the film were measured using a Hysitron TI900 Triboindenter with a Berkovich nanoindenter and 9 mN load. The hardness of the nitrided and untreated samples was evaluated with a Vickers micro-hardness tester, Shimadzu HV2 using 0.49 N loads, as the mean value of 10 measurements.

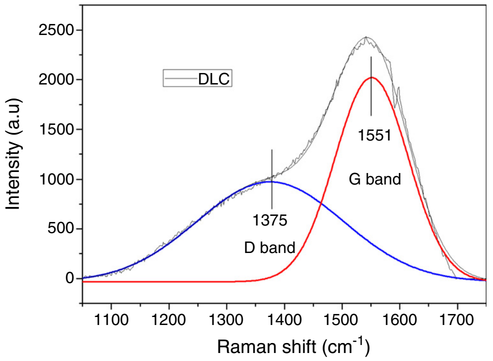

Fig. 1 – Raman spectrum of DLC coating.

Two groups of samples were coated, one was previously nitrided and the other only heat treated, named from now on, duplex and only coated systems, respectively.

The tribological behavior was evaluated by fretting and pin on disk tests. The pin on disk tests were carried out using an alumina ball of 6 mm diameter with 5 N load, in a rotational relative motion. The track radius was set at 7 mm, the tangential velocity at 10 cm/s and the total wear length, in 500 m. Fretting tests were carried out in a self-made oscillatory machine with a frequency of 23 Hz and amplitude of 80 micrometers, using also an alumina ball, 6 mm in diameter as counterpart. The tests were conducted in different load conditions: 4 N, 8 N and 12 N. Three testing times were chosen, 30, 45 and 60 min. The fretting wear tracks were analyzed by means of WLI 3D profilometry (White Light Interferometer) and were also observed by SEM. The volume was calculated considering the wear track as the half of an ellipsoid.

The adhesion was characterized using Rockwell C indentation and scratch tests. In the Rockwell C indentation tests 1500 N load was used. The scratch tests were performed with variable loads starting with 1 N and a load increasing rate of 10 N/mm. A diamond tip of 200 μm radius was used over a total distance of 8 mm. The critical load was defined as the load at which the delamination of the coating was detected. Moreover, scratch tests with a constant load of 10 N were also performed, using a diamond tip of 200 μm radius. The scratch test tracks were analyzed with a mechanical profilometer and were observed by OM. The cutting efficiency factor (fab), which considers the relation between lateral ridges area and cross sectional area of the groove, was assessed for the track profile in the coated samples [21].

3. Results and discussion

3.1. Characterization of the coating and nitrided layers

DLC coatings were characterized by Raman spectroscopy, where D and G bands were identified in the spectrum (Fig. 1). The I_D/I_G ratio was about 0.5. Taking this value into account,

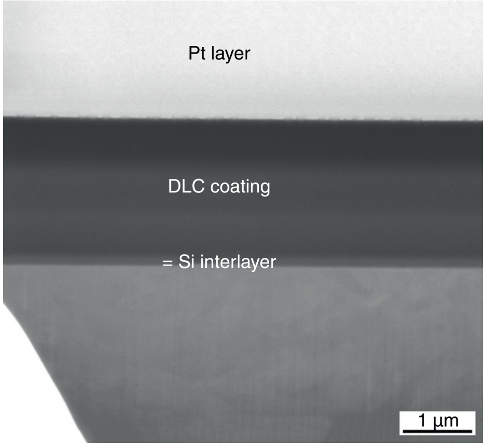

Fig. 2 – SEM-FIB image of the coated sample.

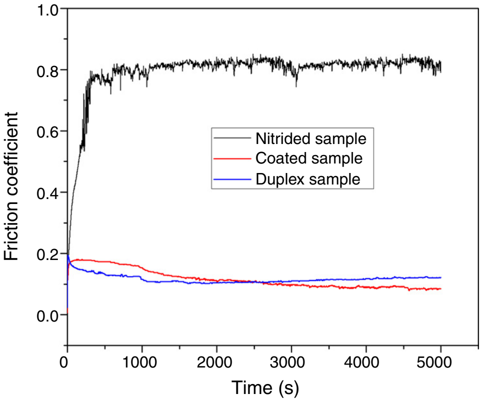

Fig. 3 – Friction coefficients registered in real time in the pin-on-disk tests for duplex, coated and nitrided samples.

the G band position and the three-stage model proposed by Ferrari et al. [22], it can be indicated that this film has around 20% sp^3 bonds. The hydrogen content was about of 20%, which was estimated from the slope of the fitted line to the base of the original spectrum [23].

The films were 2.5 µm thick with an interlayer of about 0.3 µm, as can be observed in the SEM-FIB image of a coated sample (Fig. 2). The coating presented a well-defined interphase with the substrate, both in duplex and coated samples.

The nitrided layer thickness was about 11 µm, which was measured using the optical microscope (micrograph not shown) after etching the cross section with Vilella reagent. This layer corresponds to nitrogen supersaturated martensite which is called "expanded martensite" [1,24] as it was detected by XRD and reported in a previous work [25].

The hardness of the DLC was assessed in (16 ± 8) GPa. This value corresponds to the true coating hardness because the indentation depth did not exceed 10% of the film thickness [26]. The Young's Modulus resulted in (114 ± 28) GPa.

On the other side, the nitrided layer hardness was (1150 ± 30) HV and the non-nitrided sample, only quenched and tempered stainless steel hardness was (530 ± 40) HV.

3.2. *Wear behavior*

During the pin on disk tests, the steady state value for the friction coefficients were 0.085 and 0.120 for coated and duplex samples, respectively. These values are similar to others previously reported in the literature [7,8]. The steady values were reached after 1500 s for duplex samples and after 3000 s for coated samples as it is shown in Fig. 3, which corresponds to the real time registration of the friction coefficient during the test. The friction coefficient was, on the other hand, about 0.8 for nitrided samples, which was reached 600 s after the beginning of the test. It can be observed that the friction coefficient was reduced noticeably with the presence of the DLC coating, because it is known that a transfer layer with graphitic characteristics is formed. This layer works as a solid lubricant between the coating and the counterpart reducing the friction coefficient [27–29]. With respect to the wear rate, it could not be calculated because the wear track was undetectable in the coated samples. On the other hand, the counterpart remained clean after the test, indicating that no material transfer occurred during the test in the coated samples.

Fretting tests with 4 N load and 1 h long were carried out in coated, duplex, nitrided and untreated samples. Wear volumes losses and wear rates are presented in Table 1.

The wear rate in the only coated and the duplex samples was two orders of magnitude lower than the wear rate in the nitrided or untreated samples, as it can be deducted from the values in Table 1. The depth of the wear track for the nitrided samples reached about 8 µm (and this value corresponds to 73% of the nitrided layer thickness); on the other hand, the depth of the track was approximately 0.5 µm in the coated samples. The DLC coating improved the wear resistance noticeably under this test conditions. In this case, the counterpart was clean, without material transfer in the coated samples, as it could be observed under the optical microscope after the test.

In the duplex and coated samples, more fretting tests were performed with different loads and durations in order to compare the tribological behavior of both systems (nitrided layer + DLC, steel + DLC). The results are presented in Fig. 4. In

Table 1 – Fretting test results for different samples with 4 N load, 1 h, 26.5 m wear length.

Samples	Loss volume ($\times 10^{-6}$ mm^3)	Absolute error ($\times 10^{-6}$ mm^3)	Wear rate ($\times 10^{-8}$ mm^3/N m)	Absolute error ($\times 10^{-8}$ mm^3/N m)
Coated	5.19	0.33	4.90	0.31
Duplex	4.77	0.50	4.50	0.47
Nitrided	680	74	642	70
Untreated	1053	25	993	24

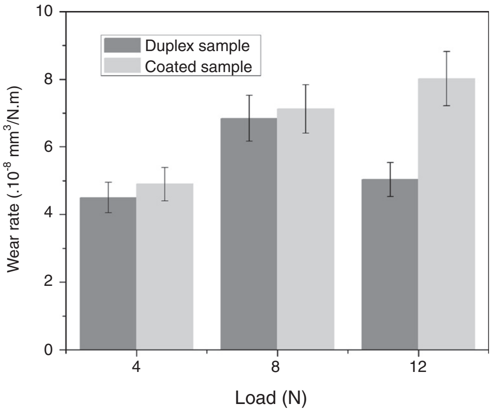

Fig. 4 – Wear rate in duplex and coated samples for tests with 4 N, 8 N, and 12 N loads.

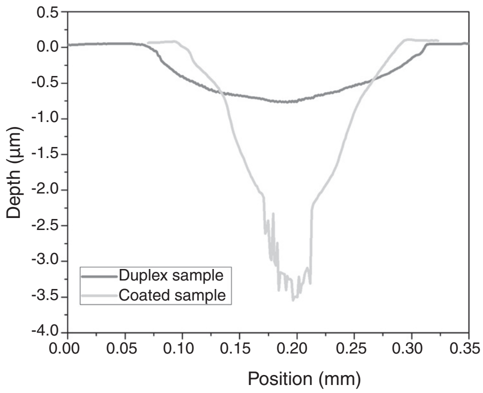

Fig. 6 – Profile of fretting wear tracks in the duplex and coated samples for 12 N and 1 h test.

the first batch of experiments, the tests were performed with 4 N, 8 N and 12 N and the duration was fixed at 1 h.

The wear behavior was similar for both samples (duplex and coated) when using 4 N and 8 N loads, which indicates that the wear resistance was mainly determined by the coating. The wear track depth was about 0.5 μm. This value did not reach 25% of the film thickness and it could be assumed that the influence of the substrate (nitrided layer in the duplex samples) was not noticeable.

With respect of the morphology, this was similar for the duplex and coated samples in the 4 N and 8 N loads tests, only some smooth grooves in the sliding direction and not very deep, could be observed in the SEM image and the WLI surface map corresponding to the 4 N load track in the coated sample, Fig. 5.

As it can be also observed in Fig. 4 for the coated sample, when load increases, so does the wear rate. The difference in the wear behavior between both samples (coated and duplex) was revealed in the experiment with 12 N load, as it can be observed in Figs. 4 and 6, where the wear track profiles are depicted. The track depth in the coated sample can be measured and it is easy to see that it is beyond the film thickness (which it was 2.5 μm), therefore the wear rate increased because part of the wear damage is produced in the substrate material, which is steel, only quenched and tempered. It can also be noticed that the wear rate was always lower in the duplex sample than in the coated sample. At this high load, the stresses distribution makes the difference, because they extend deeper and now the nitrided layer has influence in the mechanical behavior of the system, improving its resistance and reducing its wear rate.

The contact zone in the fretting tests is usually divided into two parts: a central area (without relative motion) and an annular area where microslip occurs [30] as it can be observed in Fig. 7, corresponding to a coated sample in a 1-h–12 N load test. It can be observed that the coating failed and it was detached in the central region of the fretting track. Scratches were also observed in the movement direction of the track in the enhanced SEM image, where part of the coating peeled off.

With respect the counterpart, material transfer could be detected in the 8 and 12 N load tests as it can be observed in Fig. 8, where adhered wear particles were detected after the tests for duplex and coated samples (picture obtained with the optical microscope and converted to gray scale).

Keeping the load constant at 12 N, two more tests were carried out with different durations. Showing these results together with the 12 N case of Fig. 4, it can be seen that the wear rate increases with test duration (Fig. 9). Besides, it was observed that the wear track depth was lower than the coating thickness for all tests in the duplex sample. On the other hand, in the coated sample, in the 60 min test, the wear track depth reached the substrate, and it also represents here the highest wear rate of all tested samples.

The morphology of the wear tracks was similar in the duplex and coated samples for 45 and 30 min tests, only smooth scratches in the sliding direction could be detected in the SEM images and the WLI map. Fig. 10 shows one example corresponding to the 45 min, 12 N case for the coated sample.

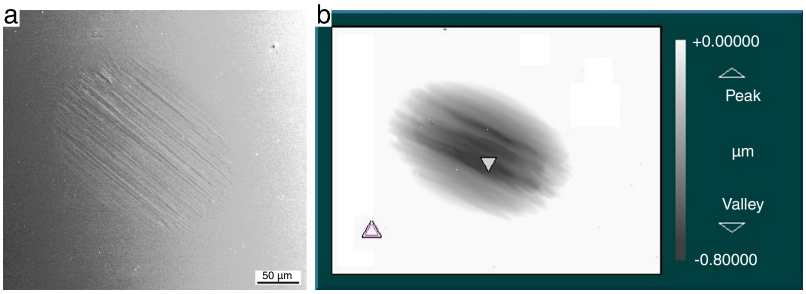

Fig. 5 – (a) SEM image and (b) WLI map for 4 N and 1 h test in the coated sample.

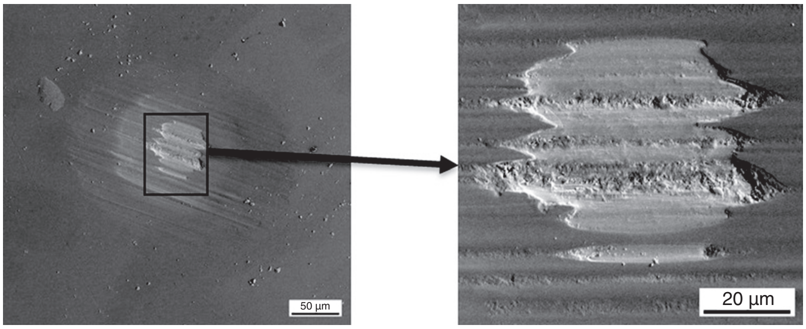

Fig. 7 – SEM images of fretting track in coated sample for 12 N and 1 h test.

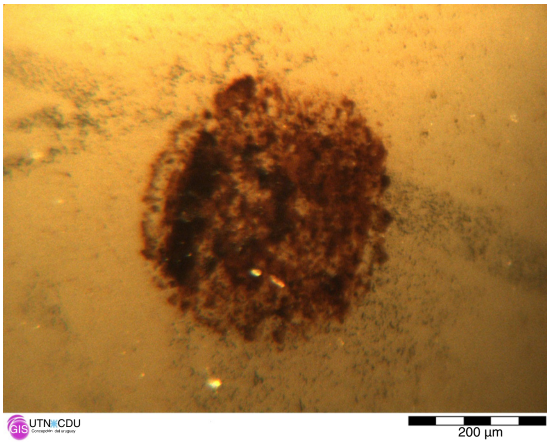

Fig. 8 – Optical micrograph of the contact zone of the counterpart after the 12 N, 1 h test for a coated sample.

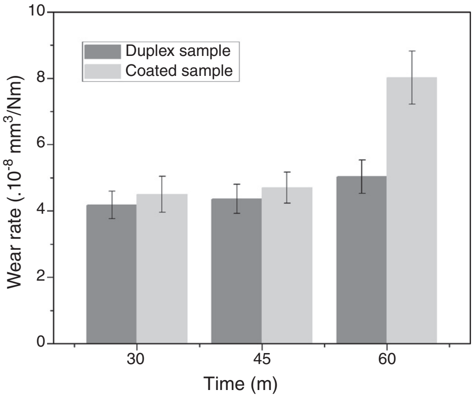

Fig. 9 – Wear rate in duplex and coated samples for tests with 30 min, 45 min and 60 min duration.

With respect to the counterpart, no transferred material was detected on the balls after the tests in both types of samples.

From the results presented in this section, it can be inferred that the wear behavior depends on the load-time combination of the tests parameters.

According to the literature, in this type of tests such as fretting or in pin on disk contact geometries on DLC films, the wear behavior is determined by the formation of the graphitized layer between the two bodies and acting as a transfer layer. As a consequence, the coating slides then on this layer which is self lubricating [30]. Probably is this phenomenon which occurred in the low load tests and with short duration, where it can be assured the wear damage was produced within the coating. However, this theory failed in the tests between 126,000 and 168,000 cycles using 12 N load. Other authors have reported approximately the same number of cycles for this kind of coating failure [31]. Probably, high residual stresses could be generated in the films (having more than 2 µm thickness) when it is deposited on soft substrates. Therefore, these stresses can be cause of the fracture and subsequently delamination of the coating as it was indicated in the literature for this kind of wear situations [5]. In this case, this could be confirmed because it was determined that the coating underwent an adhesive failure. The wear resistance of the samples could then be related to the coating adhesion [4].

The duplex system (nitrided layer + DLC) presented in all tests better wear resistance than the single system, the

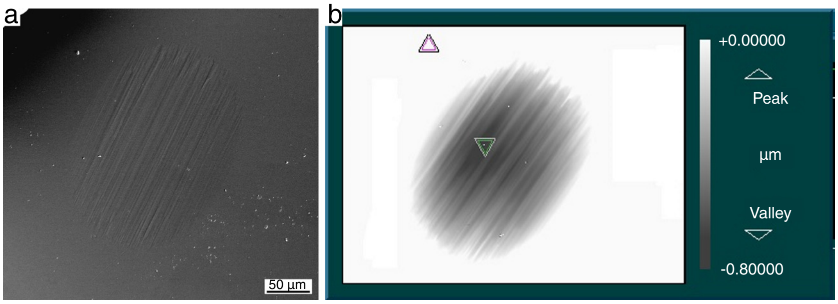

Fig. 10 – (a) SEM image and (b) WLI map for 12 N and 45 min test in a coated sample.

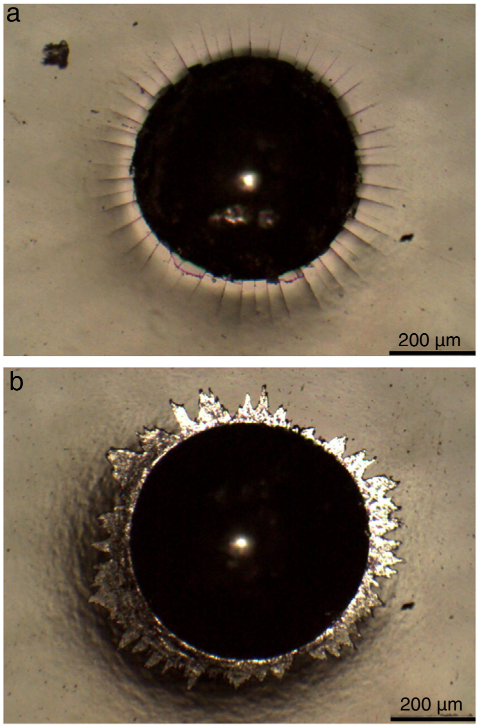

Fig. 11 – Optical micrograph of indentation Rockwell C with 150 kg load (a) duplex sample and (b) coated sample.

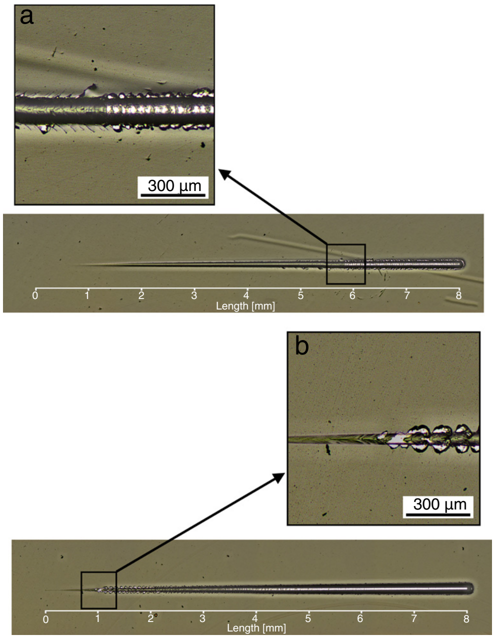

Fig. 12 – Optical micrograph of scratch test with variable load, (a) duplex sample and (b) coated sample.

coated sample. The nitrided layer improved the adhesion and reduced the stresses in the film, preventing the coating failure [15,32].

3.3. *Adhesion*

Coming specifically to the adhesion tests, it can be seen in Fig. 11a that the duplex sample presented had acceptable adhesion for a 150 kg load, although some cracks could be observed around the indentation. On the other hand, a detached region around the indentation could be seen in the coated sample in the optical micrograph (Fig. 11b). As in this test, the indenter penetrates into the coating inducing massive plastic deformation in the substrate and causing the coating fracture. It can be concluded that the nitrided layer improved the load carrying capacity and prevented film deformation and fracture, resulting in a better adhesion for the duplex system [32,33].

In the scratch test, the adhesion revealed better in the duplex sample than in the coated sample as well. The critical loads resulted in 58 N (Fig. 12a) and 12 N (Fig. 12b) for the duplex and the coated sample, respectively. Moreover, a total detachment of the coating can be observed in the coated sample.

The failure mode observed in the micrographs can be qualified as buckling spallation for the duplex sample because irregularly-spaced arcs of the coating open along in the direction of scratching. On the other hand, wedging spallation seemed to have happened because of the coated sample, since regularly-spaced and shaped, annular circular marks that extend beyond the edges of the groove can be seen [34,35]. These results indicate that the first case represents a ductile failure mode and the second, a brittle failure mode [36].

It was proposed by some of the authors in a previous work [22] that the nitrided layer improves the film adhesion not only because it reduces the stresses between the coating and the substrate but also because there is chemical affinity between the silicon present in the interlayer and the nitrogen in the nitrided layer. So it is possible that the silicon reacted with the nitrogen producing a strong chemical bonding between the substrate and the film [25].

In the scratch tests using a constant load, the coating detached at 10 N in the coated sample, as it is shown in Fig. 13a, meanwhile in the duplex sample the scratch test track was almost undetectable (Fig. 13b).

In the scratch test, the cutting efficiency factor “fab”, could be determined for the coated sample using the wear track profile (Fig. 14). It was not possible in the duplex sample because the track was almost undetectable. Fab was 0.896, and this value is close to one which indicates that the material has low ductility and toughness, according to which was reported previously in the literature [21]. The cross sectional area of the lateral ridges of the groove resulted smaller than the cross sectional area of the groove, which means that part of the coating was removed from the groove and the other part was displaced from the groove forming lateral ridges [21].

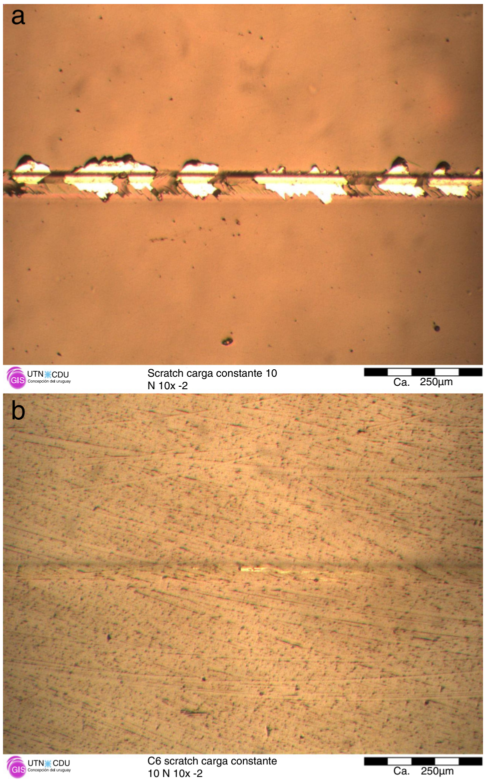

Fig. 13 – Optical micrograph of scratch tests for 10 N, (a) coated sample and (b) duplex sample.

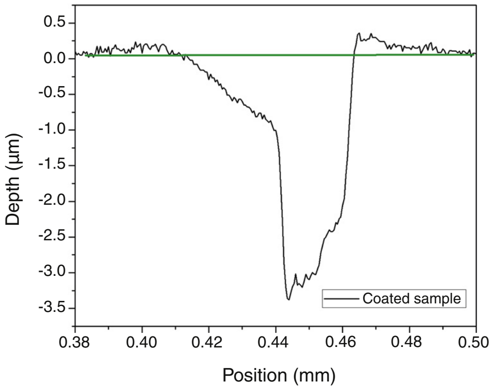

Fig. 14 – Profile of scratch test track with constant load of 10 N for the coated sample.

4. Conclusions

The DLC coatings had high hardness and low friction coefficient, as it was demonstrated in the pin on disk tests. Duplex and coated samples presented good fretting wear behavior for low loads and short duration tests. However, only the duplex samples showed good fretting wear behavior for a high load and long duration tests. The wear resistance was related to the film adhesion and the mechanical properties of the tribosystem. Adhesion proved to be acceptable only with plasma nitriding as previous treatment (duplex system) as it was revealed in the scratch test and Rockwell C indentation test, because the nitrided layer reduces the stresses and improves the load bearing capacity of the system. Moreover, the nitriding caused a modification of the coating failure mode in the scratch test, which was transformed from brittle to ductile in the duplex sample.

It was demonstrated that when the stress distribution generated in the fretting wear tests are deep enough to overcome the film, the nitrided case can be a suitable mechanical support, avoiding film delamination.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgements

The authors would like to thank Dr. Eng. M. Agustina Guitar (Saarland University, Saarbrücken, Germany) for helping with the SEM studies. We are also thankful to the students of GIS-FRCU for their collaboration with the preparation of the samples. E.L. Dalibón, N. Pecina and S.P. Brühl thank especially to the National University of Technology (Faculty of Concepción del Uruguay), Argentina, for the financial support.

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