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Abstract

Thick and soft DLC coatings were deposited over stainless steel to improve their surface properties, protecting them from wear in different conditions. When a protective film is thick, it can be considered as self-sustaining in terms of load-carrying capacity during wear situations even if the substrate is soft or hard. Thin and hard DLC coatings are well known for having high hardness and low friction coefficient; however they have adhesion problems when deposited on soft steels and many of their wear properties also depend on adhesion. For this reason, a previous plasma nitriding process may be convenient. In this work, the fretting and erosion wear behaviour of DLC soft coatings deposited on nitrided and non-nitrided austenitic stainless steel was evaluated using high loads and long tests both simulating severe conditions wear. The aim is to analyse under which conditions the film thickness is not enough to withstand wear damage when deposited onto soft steels. The results showed that in the fretting tests, the duplex sample presented better resistance than the only coated sample in all tested conditions, except for the minimum load, 12 N. In slurry erosion tests, the mass loss was similar in both samples until nine hours, when the influence of the nitrided layer became noticeable as a hard layer since the coating partially removed.

Keywords	Thick DLC, soft carbon coating, severe wear, erosion, fretting
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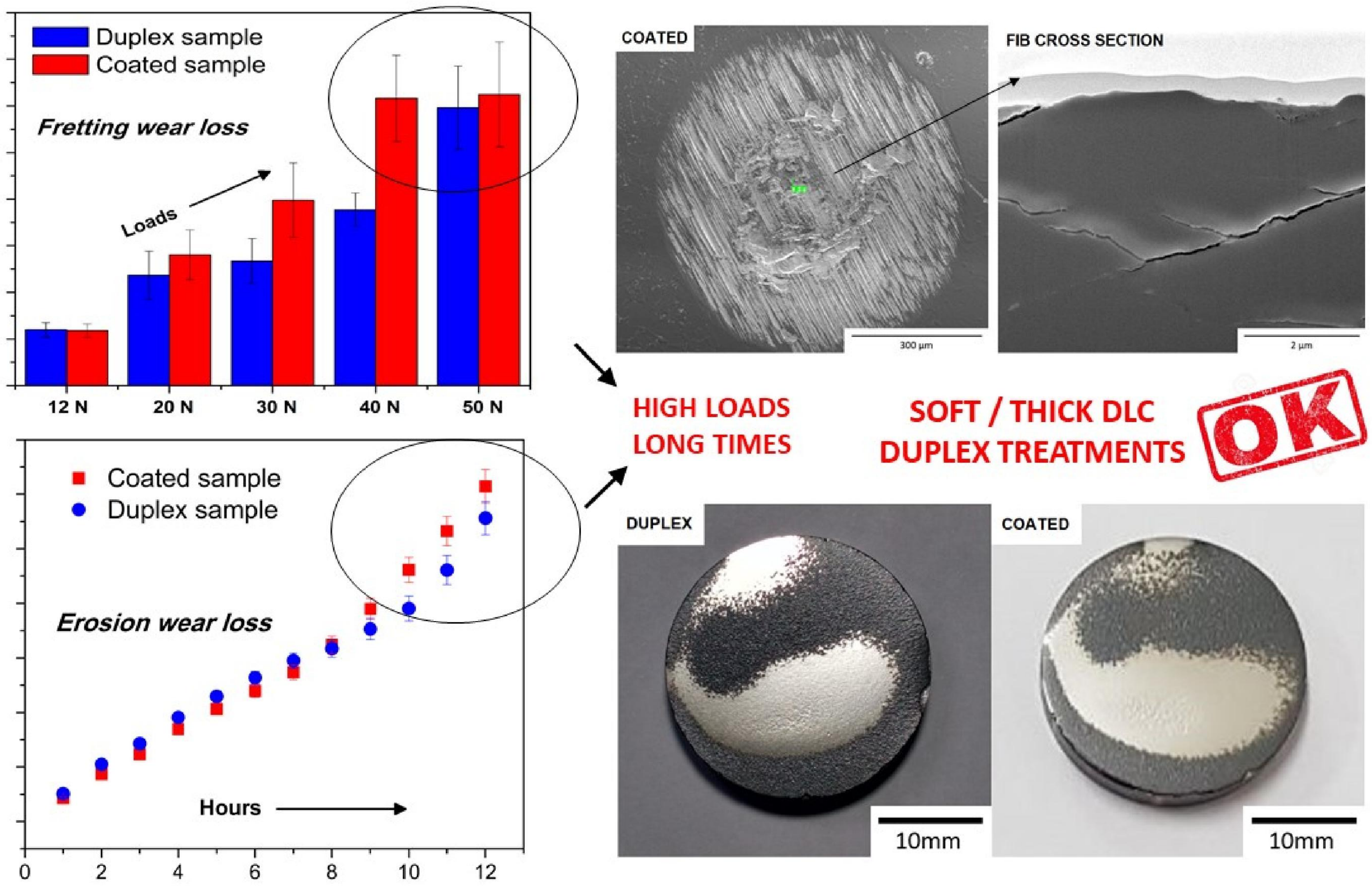
Highlights

- Soft and thick DLC films have good response to fretting wear under low loads, disregarding the pre-treatment.
- The influence of the nitrided layer became noticeable under high loads fretting tests.
- The nitrided layer was an effective mechanical support in long duration erosion tests.


Duplex sample
Coated sample
Fretting wear loss
Loads
12 N
20 N
30 N
40 N
50 N
Coated sample
Duplex sample
Erosion wear loss
Hours
0
2
4
6
8
10
12
COATED
300 µm
FIB CROSS SECTION
2 µm
HIGH LOADS
LONG TIMES
SOFT / THICK DLC
DUPLEX TREATMENTS
OK
DUPLEX
10mm
COATED
10mm

Soft and thick DLC deposited on AISI 316L stainless steel with nitriding as pre-treatment tested in severe wear conditions

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Abstract

Thick and soft DLC coatings were deposited over stainless steel to improve their surface properties, protecting them from wear in different conditions. When a protective film is thick, it can be considered as self-sustaining in terms of load-carrying capacity during wear situations even if the substrate is soft or hard. Thin and hard DLC coatings are well known for having high hardness and low friction coefficient; however they have adhesion problems when deposited on soft steels and many of their wear properties also depend on adhesion. For this reason, a previous plasma nitriding process may be convenient. In this work, the fretting and erosion wear behaviour of DLC soft coatings deposited on nitrided and non-nitrided austenitic stainless steel was evaluated using high loads and long tests both simulating severe conditions wear. The aim is to analyse under which conditions the film thickness is not enough to withstand wear damage when deposited onto soft steels. The results showed that in the fretting tests, the duplex sample presented better resistance than the only coated sample in all tested conditions, except for the minimum load, 12 N. In slurry erosion tests, the mass loss was similar in both samples until nine hours, when the influence of the nitrided layer became noticeable as a hard layer since the coating partially removed.

Keywords: Thick DLC, soft carbon coating, severe wear, erosion, fretting

1. Introduction

Austenitic stainless steels are used in different applications where good corrosion resistance is required. However, these steels show poor mechanical properties when they are used in severe wear conditions [1]. In order to improve wear resistance, different coatings and surface modification treatments can be used. There are different types of DLC ("Diamond Like Carbon") or carbon coatings and among them, the soft DLCs, amorphous and highly hydrogenated carbon coatings with thickness above 20 µm [2,3]. Besides, other elements such as F, Si, N can be added in order to modify its properties (thermal stability, electrical resistivity and surface energy), and to reduce the intrinsic stresses, allowing to grow films with greater thickness. Although there have been several publications about the wear properties of DLC coatings, there are quite a few about thick and soft DLC coatings [4–6]. As all types of carbon coatings have adhesion problems when they are deposited directly on steels, some treatments or interlayers were used in order to improve adhesion, thus reducing interface stresses. Also some hybrid or duplex systems (nitriding plus hard-thin DLC coating) on stainless steel have been studied [7–10]. It was determined that the nitrided layer properties have a clear influence on the mechanical behaviour of the nitrided steel-DLC coating system, as well as on the corrosion behaviour [8,11,12].

Since the deposited DLC thickness is over 35 µm, the influence of the nitrided substrate will be studied. A thick film can withstand wear regardless the substrate because the stress distribution in a certain wear situation might not reach the interface, as it does in a thin film. In previous works, sliding wear experiments with different loads, both abrasive and erosion wear tests were carried out on thick DLC deposited with PACVD (Plasma Assisted Chemical Vapour Deposition) over nitrided AISI 316L [13–15]. In this work, the same duplex system (soft DLC + nitrided layer) will be tested under erosion conditions (although this time more severe) and under fretting wear. This system will be compare with the same coating deposited onto the bare steel without nitriding as pre-treatment. The influence of the nitrided layer will be studied using different loads in fretting tests, and in slurry erosion tests until twelve hours are reached.

2. Materials and Methods

Commercial AISI 316L austenitic stainless steel was used as base material. The samples were obtained from a steel bar of 25 mm in diameter, which had been sliced to obtain 6 mm discs. The material chemical composition is 0.017 wt.% C, 0.33 wt.% Si, 1.44 wt.% Mn, 16.25 wt.% Cr, 10.07 wt.% Ni, 2.03 wt.% Mo, 0.24 wt.% Cu and Fe as balance.

The plasma nitriding treatments were carried out at 400ºC for 14 hours in a 20 % N_2 - 80 % H_2 gas mixture with bias voltage of 320 V and a pressure of 2 mbar in a semi-industrial hot wall reactor. The carbon coatings were deposited by means of PACVD (Plasma Assisted Chemical Vapour Deposition) in the same reactor. The system consists of a chamber with an auxiliary heating system, a gas supply and distribution system, and a pumping system with pressure control. The discharge is maintained by a pulsed DC power supply with a duty cycle of 20 % and a pulsing frequency of 1 kHz at a voltage of 330 V. The reactor is a stainless steel vessel, which acts as anode and the substrate plate serves as the cathode as it was depicted and schematized in Ref. [5]. Hexamethyldisiloxane (HMDSO) and acetylene were used as precursor gases (8% HMDSO and 92% acetylene). The processes were performed at 425 °C and a pressure of 2 mbar. The deposition rate was about 0.5 – 1 µm/h. Silicon was added in the deposition process in order to reduce the stresses and consequently the deposition of thick coatings could be achieved. As coatings grown by CVD are usually amorphous and as there are many kinds of DLC films with different dopants, it was decided in this work to name these coatings as a-C:H:Si (silicon containing amorphous hydrogenated carbon).

The samples coated with a-C:H:Si on nitrided stainless steel were named “duplex” and the samples coated with a-C:H:Si on bare stainless steel were named “coated”.

The microstructure of nitrided layers and the film thickness were analysed by optical microscopy (OM) and SEM. The a-C:H:Si structure was analysed by Raman Spectroscopy. The nanohardness was measured using a Berkovich indenter with 9 mN load and the microhardness was evaluated using a Vickers indenter with 25 g load.

The adhesion was evaluated by the Scratch Test with constant loads of 30 N, 40 N and 45 N. The scratch tracks were observed by OM and a confocal microscope.

In order to evaluate the wear resistance, fretting and erosion tests were carried out. The fretting tests were performed in a homemade machine using different loads (12 N, 20 N, 30

N, 40 N, 50 N) for 1 hour with alumina as counterpart (6 mm in diameter). The frequency of the oscillating movement in a configuration ball on plane was 23 Hz and the amplitude 80 µm. The fretting wear tracks were observed using SEM, WLI 3D (White Light Interferometer) and a confocal microscope. The volume loss was calculated using the WLI profiles, considering that the wear scar has the shape of a semi ellipsoid.

The erosion tests were conducted using a self-made erosion machine using a mixture of water and sand (AFS 50) flux that impacts with an angle of 60° at a velocity of 7 m/s. The tests lasted 12 hours but the samples were weighed every hour using an analytical balance with a 0.1 mg resolution. The mass loss was calculated as the weight difference of the samples before and after each hour.

3. Results and Discussion

3.1 Microstructure

The DLC coatings were characterized by Raman spectroscopy (Fig. 1). The two characteristic bands were detected, D and G, the intensity ratio (I_D/I_G) was 0.95 and the proportion of sp^3 C-C bonds was about 10% and H content about 43%. These coatings are similar to those which were studied by some of the authors in a previous paper [13]. In addition to hydrogen, they contain silicon, as it was reported before [13].

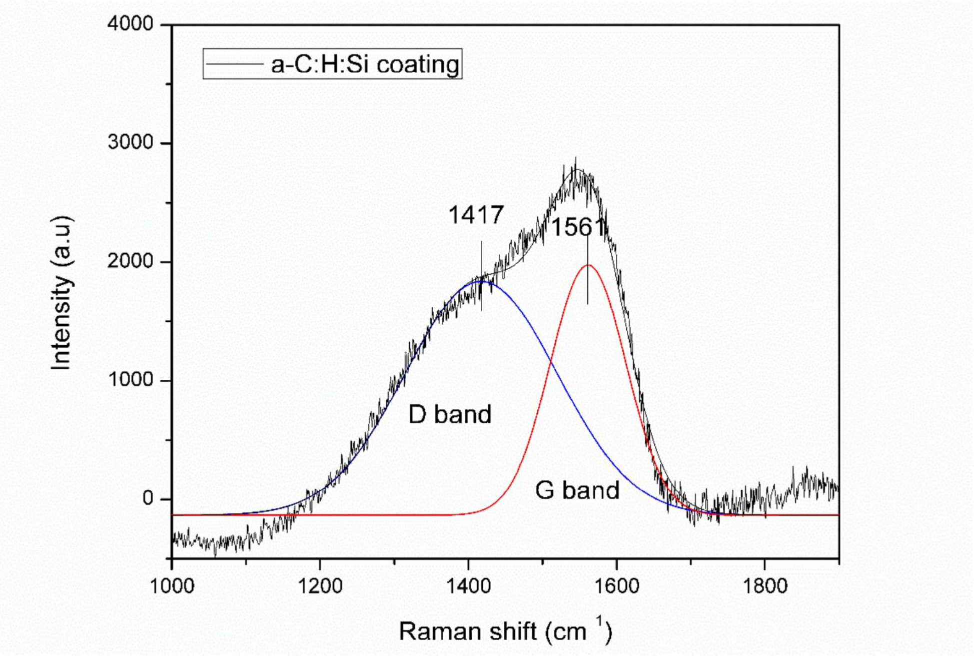

Fig. 1: Raman spectrum of the a-C:H:Si coating

The coating thickness was (38 ± 1) µm with a regular interface with the substrate (Fig. 2). Some irregularities were observed in the coating cross section and it is probable that these defects were produced during the deposition process.

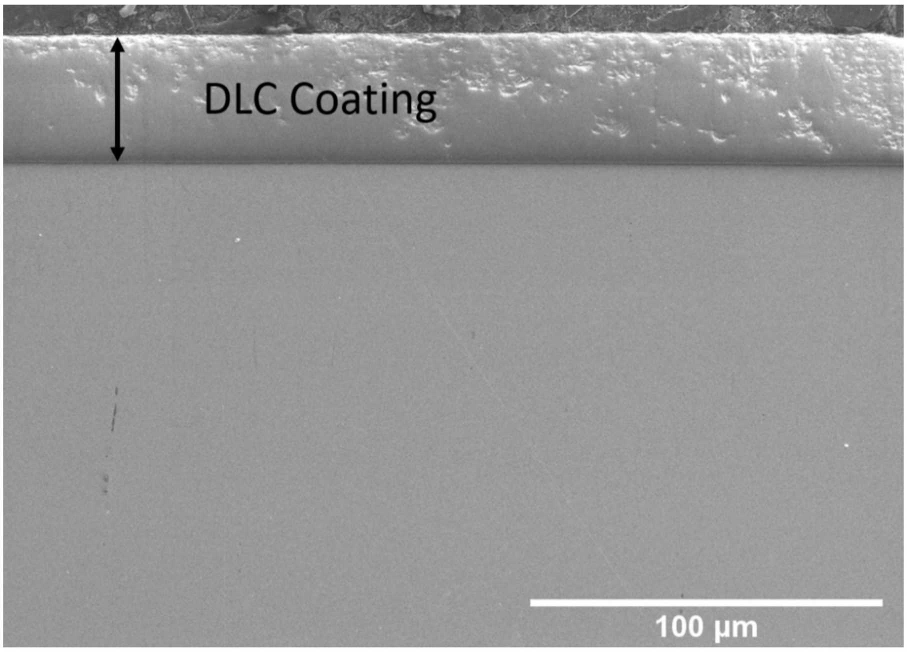

Fig. 2. SEM image of the coating for the coated sample.

The nitrided layer thickness (not shown in Fig. 2) was about 10 µm. This layer corresponds to the so called expanded austenite, a phase whose formation is well known for this kind of steels after the plasma nitriding process [16,17]. Chromium and iron nitrides were not detected by means of XRD measurements as it was reported in a previous work [13].

The a-C:H:Si coating nanohardness was (12 ± 2) GPa and the Young's Modulus was (73 ± 5) GPa. The nanohardness corresponds to the coating because the indentation penetration depth did not exceed the 10% of the coating thickness [18]. This type of DLC coating may be considered soft due to its low hardness and Young's Modulus, caused by the high hydrogen content. The nitrided layer microhardness was (900 ± 20) $HV_{0.025}$ (8.82 GPa) and the Young's Modulus was (134 ± 11) GPa. The untreated sample hardness was (250 ± 20)

HV$_{0.025}$ (2.45 GPa). The hardness after the nitriding process increased about four times compared to the untreated samples.

3.2 Adhesion

Scratch tests with different constant loads (30 N, 40 N, and 45 N) were performed. The adhesion was similar in both samples for 30 N and 40 N, but there was one difference for 45 N, where the coating detached only in some areas along the track. The damage was greater for the coated than the duplex samples as it can be observed in Fig. 3. The tracks profiles were obtained for both samples and they are shown in Fig. 3. The track depth was similar for both samples using 30 N; however, with 45 N load the maximum depth was higher for the coated sample than for the duplex sample. This value represents 40% of the coating thickness. The load values for which the coating failed were similar or even higher than those obtained in hard DLC coatings [19,20]. In this case, the coating hardness is lower but since the coatings are thick, the stresses could be distributed more homogeneously and could not reach the interface, preventing the total coating detachment [5]. Besides, the nitrided layer improved the adhesion due to an increase the substrate hardness, causing stresses reduction and enhancing the load-carrying capacity, as it was reported in a previous publication [15].

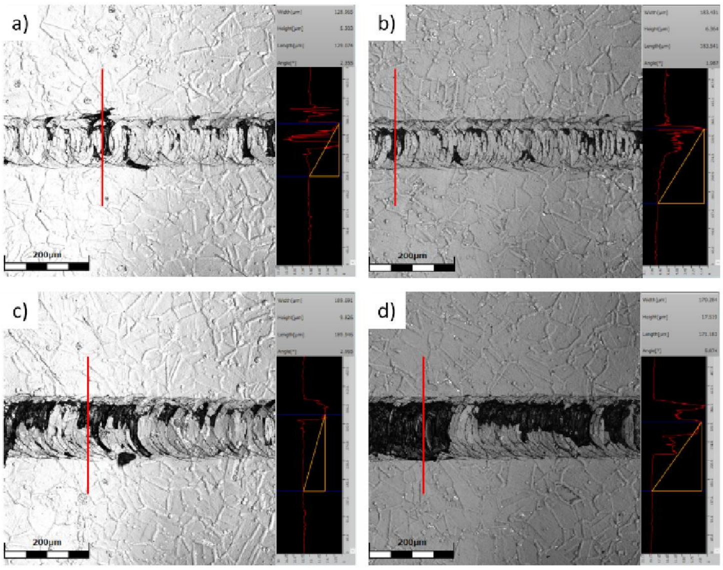

Fig. 3. Confocal images of scratch test tracks. a) and c) 30 N and 45 N load respectively for duplex sample. b) and d) 30 N and 45 N respectively for coated sample.

Regarding the failure mode for higher loads, in coated and duplex samples, this can be considered "buckling cracks", because the coating buckled ahead of the tip. Regularly spaced arcs were produced and opened away from the scratching direction [21]. However, the detached area along the track was larger for the coated sample (Fig. 3d) than the duplex sample (Fig. 3c). It is possible that the stress gradient in the coated system was higher than in the duplex system due to the relation between elastic moduli [22,23]. $E_{coating}/E_{steel}$ ratio was higher than $E_{coating}/E_{nitrided\_layer}$ and consequently the response to the adhesion test was better in the duplex sample than in the coated sample.

3.3 Wear behaviour

These a-C:H:Si coatings have been evaluated previously in pin on disk tests using Al_2O_3 as a counterpart (with maximum Hertzian contact pressure of 0.78 GPa) and they showed low steady friction coefficient (approx. 0.1) as mentioned in [13].

3.3.1 Fretting tests

The fretting tests with different loads were performed and the volume loss and wear rate were calculated. The results are presented in figures 4a and 4b.

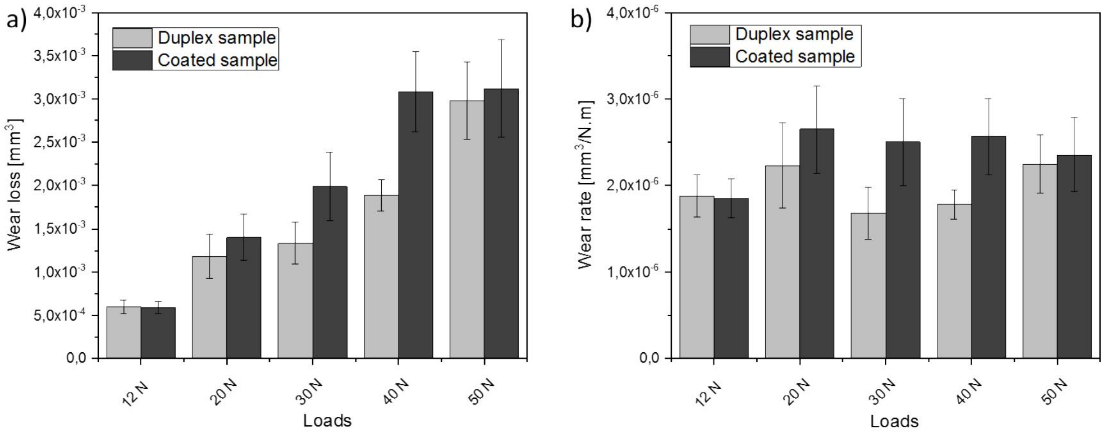

Fig. 4. Wear volume loss and wear rate for different samples in the fretting tests using 12 N, 20 N, 30 N, 40 N, 50 N load.

For 12 N load, the wear volume loss and wear rate were similar for both duplex and coated samples. Probably, the wear process occurred mainly in the coating in both samples since the track depth was about 7.5 µm. As it was expected, the more the load increased, the higher the wear loss. However, the coated samples increased the wear loss when compared with the duplex samples at the same load (Fig. 4a). At the highest load (50 N), this difference decreased.

The wear rate changed starting from 12 N to higher loads, as it can be observed in Fig. 4b. The volume loss difference between tests using 20 N and 12 N was about 49 % for duplex and 58 % for coated samples, and the scar depth increased from 7.5 µm to 15 µm for the duplex samples (Fig. 5).

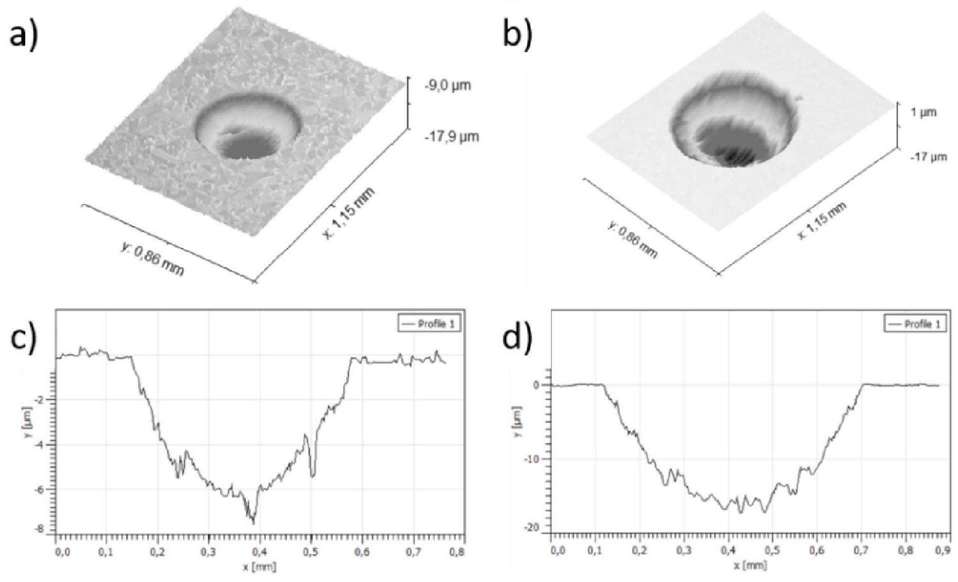

Fig. 5. Confocal images of fretting wear tracks for the duplex samples. a) and c) 3D confocal image and profile for 12 N load. b) and d) 3D confocal image and profiles for 20 N load.

Although the wear scar did not reach 50 % of the coating thickness, this change in the wear behaviour could be related to the shear stresses distribution [24]. Normally, a coating fails by detachment (adhesive failure) or fracture (cohesive failure).The adhesive failure happens when the stresses are localized in the interface and the cohesive failure, when the stresses are placed into the coating [24]. For all cases in this study, the Hertzian pressure (P_H) and shear (τ_{max}) stresses were calculated. In addition, shear stresses along the depth axis were calculated and together with the maximum values are indicated in Fig. 6. For each wear condition test, a different curve was obtained and it can be seen that the maximum shear stresses are located at about 37 μm for 12 N load and at 44 μm for 20 N load. Thus, the maximum shear stress changed from being inside the coating to being outside, just beneath it. Consequently, the load bearing ability of the system changed with the load and the wear volume loss increased significantly between wear experiments with 12 N and 20 N (Fig. 4). It could be also observed that the influence of the nitrided layer became noticeable; the increment in the wear rate was not as high as the non-nitrided one. This change in the wear behaviour evidenced that even though the coatings develop intrinsic stresses (defects and structural changes that are produced during the deposition process), the extrinsic stresses distribution resulting from the external load determined the wear resistance, for each experimental condition.

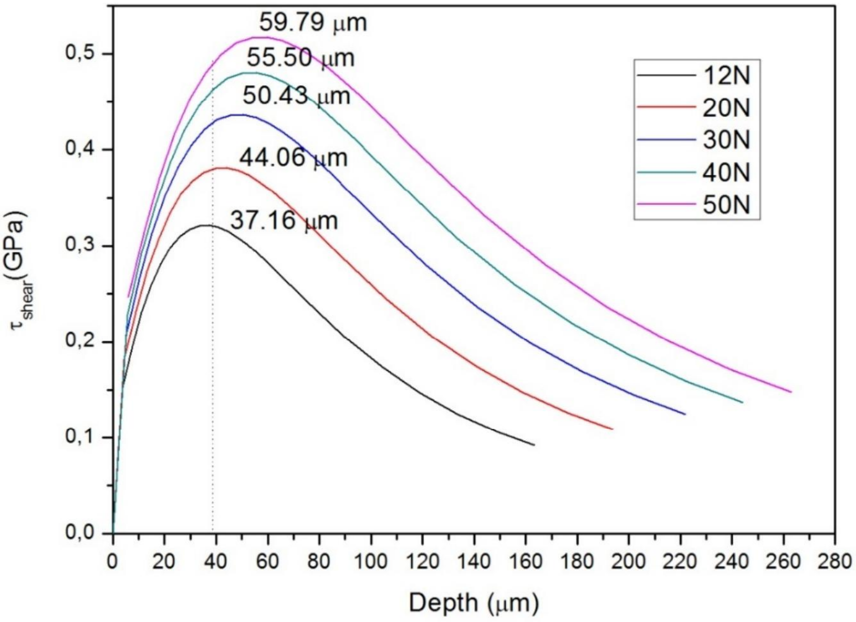

Fig. 6. Comparison among shear stresses along the depth axis for the a-C:H:Si coating and at different load conditions.

As already mentioned, for loads higher than 20 N, the wear resistance was better for duplex samples than for coated samples. This can be explained because when the load increased, the influence of the substrate became relevant. Since the H/E value is higher for nitrided layer than for stainless steel, the former becomes more resistant to plastic deformation. The duplex samples showed better wear behaviour than the coated ones. Again, the mismatch of hardness and Young´s Modulus between the coating and the substrate could affect the wear behaviour in these conditions ($E_{coating}/E_{nitrided\_layer}$ was higher than $E_{coating}/E_{steel}$) [22,23]. The difference between the wear behaviour of the coated sample and the duplex sample was not so clear for 50 N, where the shear stress maximum value falls in the steel directly and the influence of the nitrided layer was not as important as for lower loads.

Regarding the morphology of the fretting wear tracks, two zones can be distinguished; one annular and the other central, which is typical of the fretting wear damage. Grooves in the direction of the movement can also be observed in the whole track (Fig. 7, SEM image, 50 N, 1h, 82800 cycles). In addition, the central region turned out to be the most damaged due to the fact that normal pressure was higher and the wear more severe [25]. Anyway, as it was mentioned above, the coating was not completely detached because Si and C signs were detected in the EDS measurements, which were performed inside and outside of the wear scar (Fig. 7).

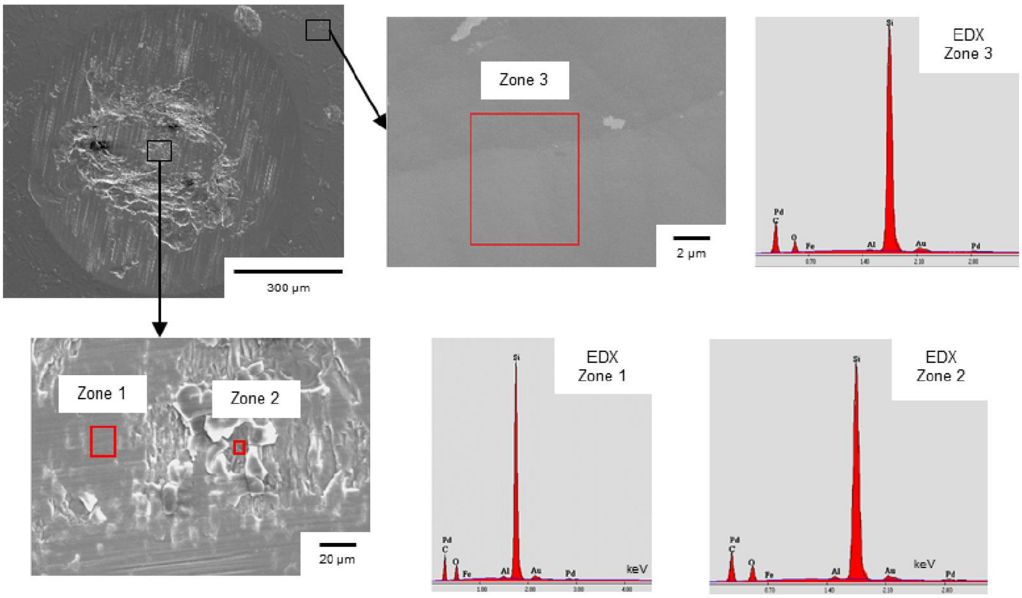

Fig. 7. SEM images of fretting wear track and EDS spectrum in different zones for the test with 50 N, 1 h.

The FIB cutting was performed across the fretting wear track for the sample tested with 50 N, 1 h with the aim to evaluate the damage under the surface (Fig. 8). As it can be observed, the cracks started in the surface, and they probably extended in the film during the 82800 cycles test (1 hour). Different cracks could connect with one another causing part of the coating to break and be pulled apart. In this case, it might be concluded that the coating suffered cohesive failure, because it was partially peeled off layer by layer.

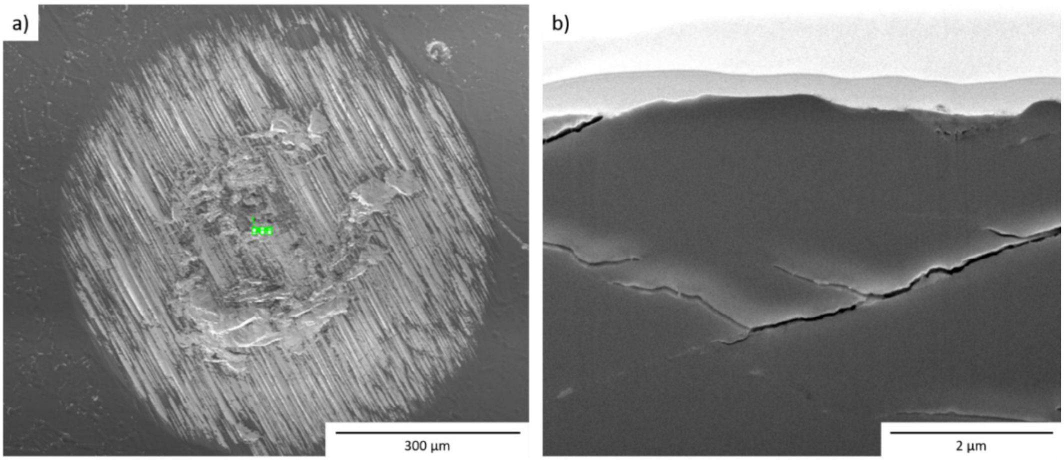

Fig. 8. a) Fretting wear track (50 N, 1 h) SEM image, b) FIB cutting in the fretting wear track.

Concerning the counterparts, transfer material was detected in practically all tests. One example can be observed in the optical micrograph of Fig. 9. As it is well-known, in tribological tests with carbon coatings a transfer layer is usually formed and it has graphitic characteristics [3,26]. This transfer layer helps to reduce the friction coefficient and thus the wear rate.

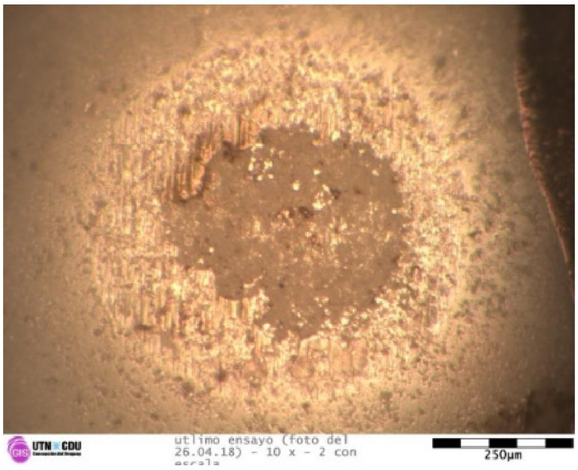

Fig. 9. Optical micrograph of counterpart for 1h and 50 N test in the duplex sample.

These a-C:H:Si soft coatings presented good wear resistance in fretting wear tests compared to the hard DLC and graphite-like carbon (GLC) films. The DLC hard coatings had low wear rate but they failed at high load (50 N) for about 15000 cycles as it was reported [27]. Meanwhile, the GLC coatings had large delamination after 6000 cycles under less severe conditions (lower Hertzian pressure) [28,29]. These a-C:H:Si soft coatings showed a different failure mode; they did not detach completely even under the most severe conditions. In fact, one experiment was conducted with the maximum load (50 N) where the duration was increased to 2 hours. Even for this condition, the wear rate remained constant and the film still resisted without total detachment (results not shown).

3.3.2 Erosion

The a-C:H:Si soft coatings improved the erosion resistance in comparison to the untreated samples. The mass loss was almost three times lower in the coated samples (with and without previous nitriding treatment) than the untreated samples (Fig. 10).

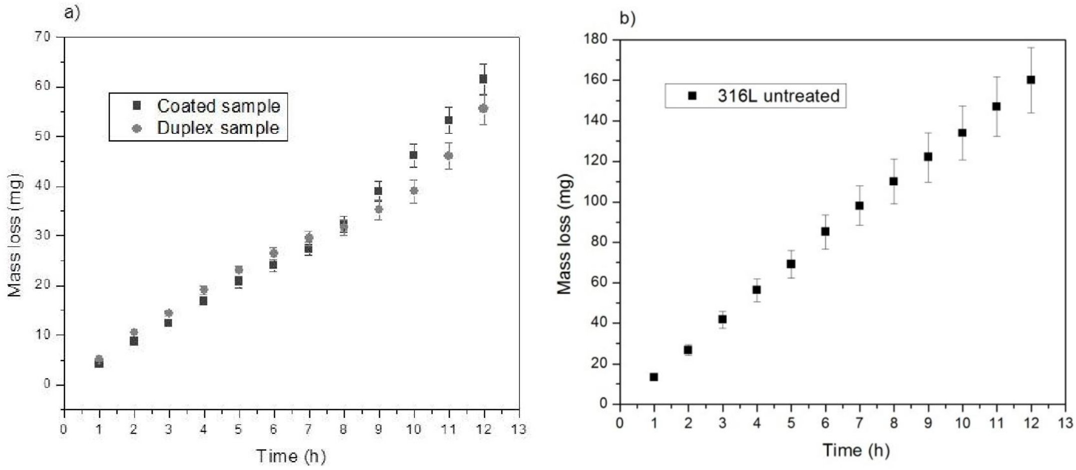

Fig. 10. Loss mass for different samples in the erosion tests for each hour. a) duplex and coated samples, b) untreated sample.

It can be also observed in Fig. 10a that the duplex and coated samples had a similar behaviour until the eighth hour since the mass loss was comparable for both. For longer tests, the mass loss was higher for the coated samples than for the duplex samples. This can be due to the spatial distribution of the particle density in this test causing non-uniform wear; the coating was detached in some areas and the substrate was exposed (Fig. 11). Consequently, the better wear resistance of the duplex sample compared to the coated sample should be attributed to the nitrided layer, which is harder and more resistant to the plastic deformation than the bare steel.

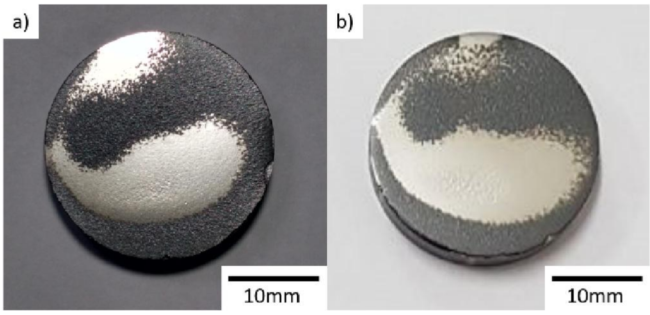

Fig. 11. Surfaces of the samples after a 9 hours erosion test. a)duplex sample, b) coated sample.

Probably, the coating was removed layer by layer in some areas. However, in others, it was completely removed (Fig. 10). It is possible that some cracks nucleated on the surface and their sizes were enlarged due to successive impacts of erosive particles. These cracks produced grooves which extended and connected with each other causing the detachment of some parts of the coating [30]. A closer look to the surface allowed determining that the substrate had suffered plastic deformation.

4. Conclusions

The soft and thick a-C:H:Si coatings were deposited on nitrided and non-nitrided austenitic stainless steels (named duplex and coated samples respectively). The adhesion test showed a high critical load for both cases, duplex and coated, but the positive influence of the nitrided layer as support layer could be observed in the extension of the damaged area along the scratch track. This was smaller in the duplex than in the coated sample. As the substrates (steel and nitrided steel) have different elastic moduli, the ratio with the coating´s modulus was higher for the duplex sample than for the coated sample, the stress gradient was consequently lower and the adhesion improved. Moreover, this had a positive influence on the wear resistance.

The coated and duplex samples had similar fretting wear resistance for low loads, but there was a marked difference for higher loads where the influence of the nitrided layer became noticeable. Although, in none of the cases the coating was completely detached, the wear volume loss was higher for the coated than the duplex sample. The change in the wear loss volume became noticeable for tests at 20 N where the maximum of the shear stress changed from being in the coating to being right under the coating, where the mechanical resistance of the system is lower.

In the erosion tests, the coatings had similar behaviour in the duplex and the coated samples until eight hours test duration. For longer tests, however, the duplex had better resistance due to the presence of the nitrided layer, as a harder substrate. This is because in the erosion test, the sand flux causes non-uniform wear, the coating peeled off and the substrate was exposed.

Even though the a-C:H:Si coating was thicker than the usual coatings, it was proved that for the high loads in all the mechanical tests carried out in this work, the nitriding treatment played its role improving the load-bearing capacity of the system. Following the aim and the results obtained in this work, it is concluded that the nitrided layer had a great influence in the wear behaviour when the tests were performed under severe conditions such as high pressure and long durations.

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