

# WEAR BEHAVIOUR OF PLASMA NITRIDED AISI 316L AUSTENITIC STAINLESS STEEL

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## ABSTRACT

Austenitic stainless steels are widely used in industrial applications, mainly due to its good corrosion resistance; however, its low hardness and poor wear performance impose strong limitations in many cases. During the last decade, plasma nitriding has gain acceptance as a technology for surface modification which seems to overcome these problems; in addition, several works carried out at laboratory scale have reported improvements on wear characteristics of plasma nitrided samples; consequently, it became of interest to study the wear behaviour of samples nitrided with an industrial equipment.

In this work we present results of wear tests performed on AISI 316L stainless steel samples nitrided with an industrial DC-pulsed plasma equipment during 20 h at 400°C in a plasma formed with 25%N<sub>2</sub>+75%H<sub>2</sub>. The wear tests were made with an Amsler-Disc-Machine A 135 under rolling-sliding conditions; the surface and subsurface were studied by optical microscope (OM) and microhardness. Wear debris generated under dry rolling-sliding condition were observed by scanning electron microscope (SEM).

Analysis and discussion of results show that plasma nitriding improves the wear resistance of the surface on austenitic stainless steel and the main wear mechanism appears to be delamination.

Consequences on the load bearing capacity are discussed.

## KEYWORDS

Plasma-nitriding, AISI 316L, wear tests.

## INTRODUCTION

Austenitic stainless steels are well known by its corrosion resistance and, as a consequence, are extensively used in chemical, nuclear and food industries. Nevertheless, they show as a principal drawback, low hardness and wear resistance, and high friction coefficient; all these can be summarized in poor tribological characteristics (1,2). In the past, different methods have been tried to modify the surface and overcome the weakness mentioned, without affecting the corrosion resistance, within that methods we find ion nitriding and ion implantation (3,4).

The industrial use of ion nitriding is growing in relation to gas and salt bath nitriding, mainly because it can be applied at low temperature and it is an environmentally friendly technology (5). Another advantage is that, being a diffusion controlled thermo chemical process, the case formed begins in the surface and reduce its thickness gradually towards the base material; this avoids problems of adherence and spalling off that can be present when a coating technology as PVD or CVD is used, mainly because of the low hardness of austenitic stainless steels. Different works have reported results of ion nitriding stainless steels within the range of 300 °C to 440 °C, the lowest temperature is the minimum value to obtain a reasonable layer thickness within an acceptable time while 440 °C is the temperature where precipitation of chromium nitride (CrN, Cr<sub>2</sub>N) begins to form on the surface and near to the surface. Once the precipitation process has started, the corrosion resistance become impaired (5-7).

Other works (8,9) have studied the effect of sliding wear on austenitic stainless steels and have found that the amount of debris is proportional to the travel distance during the test, showing a debris with a great proportion of  $\alpha'$ -martensite for the loads applied (8N – 48N). Some researchers (9) have found that the wear and friction behaviour of these steels depends on their stability in relation to the transformation of austenite to  $\epsilon$  (h.c.p.) and  $\alpha'$ (b.c.c.) phases, nevertheless it is necessary to do more work for a better understanding of this phenomena.

On this work we present wear results and a microstructure analysis of an ion nitrided austenitic stainless steel AISI 316L. The wear tests were performed in the rolling-sliding condition under different loads.

## EXPERIMENTAL WORK

The steel used had the following chemical composition (wt - %): 0.02C; 1.53 Mn; 0.023 P; 0.024 S; 0.69 Si; 16.4 Cr; 10.4 Ni; 2.00 Mo; balance Fe. Its microstructure was fully austenitic with some small manganese sulfide inclusions and its hardness was  $\sim$  160 HV. Samples were prepared as discs, 40 mm diameter and 10 mm thickness machined from annealed bar, after machining samples were ground and polish with emery paper to grade 600. A total of 40 samples were prepared, 36 were nitrided and the remainder used as reference in the wear tests.

The ion nitriding process was performed using an industrial equipment with a DC pulsed power supply, and a chamber of 600 mm diameter and 1200 mm high; temperature was measured with a thermocouple in contact with the samples, the specific conditions are presented in Table I.

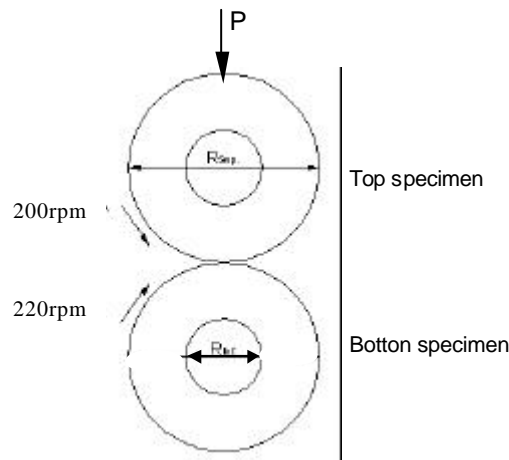
Parameter	Value
Time	20 h
Temperature	400 °C
Pressure	6.5 hPa
Atmosphere	75 % H <sub>2</sub> + 25 % N <sub>2</sub>
Voltage	700 V
Duty Cycle t <sub>on</sub> /t <sub>off</sub>	70-200 μs
Current density	~ 1 mA.cm <sup>-2</sup>

**Table I:** Nitriding conditions

Before nitriding, in order to remove the oxide film from the surface, the samples were subject to an sputtering process during 3 hours using a gas mixture of 50 % Ar and 50 % H<sub>2</sub>.

The preparation of samples for metallography was made by cutting them parallel to the rotation plane (longitudinal) and transverse to it, polishing up to grade 1μ and finally using Marble reagent for development of microstructure features.

The wear test were made with an Amsler machine model A 135 under de rolling-sliding condition (10 % sliding) under 50, 125 and 200 kg loads. The arrangement of samples is presented in the Fig.1 where the top sample rotated at 200 rpm and, to obtain 10 % sliding, the bottom sample rotated at 220 rpm. Three sets of two samples, were used for each load.



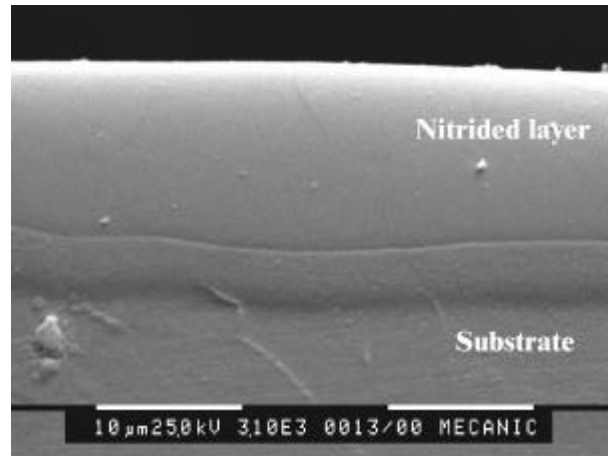
**Fig. 1:** Scheme of samples in the wear machine

During the test, periodic interruptions were made in order to measure the weight loss and to recover the debris for its analysis. After each interruption, the samples were cleaned with alcohol and acetone using an ultrasonic bath; the debris recovered and the samples, were weighted with a Mettler Toledo AB204 (0.0001g) balance. When restarted the test, samples were mounted in the same position as before. Samples and debris were observed by optical microscopy (OM) using a Zeiss Axiotech equipment and by scanning electron microscopy (SEM) using a Philips SEM 505 equipment. The microhardness measurements were made on transverse sections of the samples using an equipment Akashi MVK-H2.

## RESULTS AND DISCUSSION

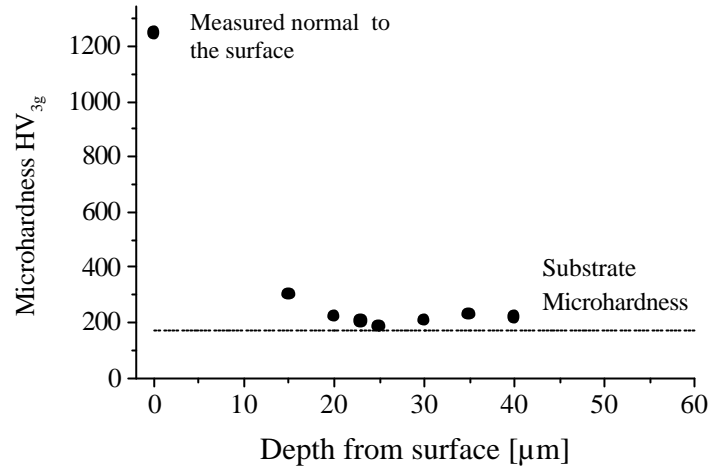
### Microstructure and Microhardness

Fig. 2 shows a scanning electron micrograph of a cross section of one sample nitrided under the conditions of Table I. It can be observed the nitrided layer and the transition zone to the substrate. The layer appears uniform and continuous without dark zones characteristic of nitride precipitates (10). It can be seen that the layer is formed by two sub layers being its total thickness around 15  $\mu\text{m}$ .



**Fig. 2:** Scanning electron micrograph of a sample nitrided at 400 °C during 20 h; it can be seen the nitrided layer formed by two sub layers.

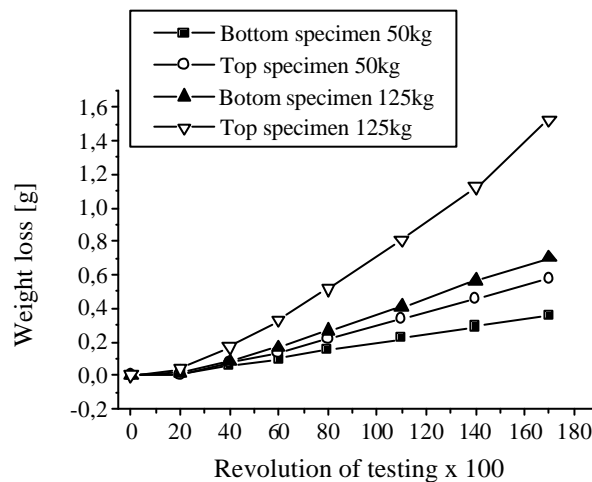
Different works report that, when the nitriding process is done between 310 °C and 420 °C, the layer become formed by a super saturated solid solution of nitrogen in iron  $\gamma$  (f.c.c.) also called expanded austenite ( $\gamma_N$ ). This phase presents high hardness and very good corrosion resistance (10-12). In the review work of T. Czerwiec et. al. it is also reported the presence of two layers, each one with different nitrogen concentration, the higher level corresponding to the more external, in contact with the plasma. They suggest that the formation of the two layers takes place during the slow cooling after nitriding (12). Besides this, K. Marchev et. al. have suggested that the layer microstructure is body center tetragonal (b.c.t.), called '*phase m*' (5) which do not agree with Thomas Christiansen, who nitriding samples of sintered steel, concludes that the phase is f.c.c. (13). Other authors (14) also encountered a double layer, but the second one was carbon expanded austenite, where carbon was taken from the reactor walls, which was previously used in a DLC- deposition CVD experiment. These composition and structural aspects will be not be discussed in this work. Fig. 3 shows a typical microhardness profile of a sample cross section. It can be observed the sharp transition from the high hardness in the surface to the low hardness of the substrate, this transition is associate to the presence of the  $\gamma_N$  layer (10).



**Fig. 3:** Microhardness profile of a sample ion nitrided at 400 °C during 20 h

### Wear

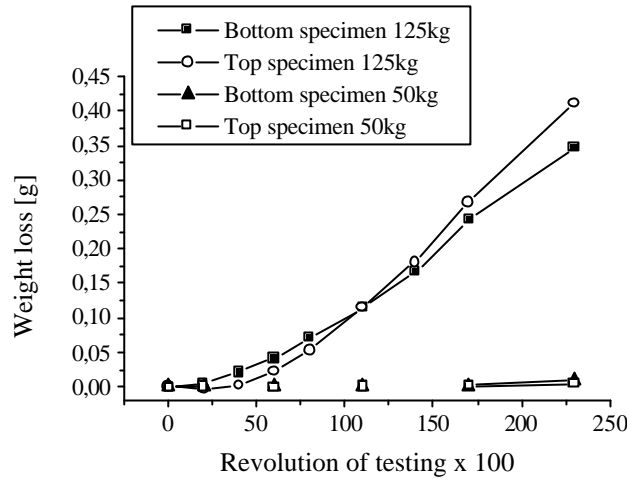
Fig. 4 presents results of weight loss as a function of number of revolutions for samples tested in the as received condition (no nitrided) under the rolling-sliding condition (10 %), with 50 and 125 kg loads.



**Fig. 4:** Weight loss of samples as received (no nitrided) as a function of revolutions.

The weight loss of the as received samples, tested at 50 and 125 kg load appears approximately proportional to the number of revolutions. Nevertheless, the wear was systematically greater on the top samples. The behaviour observed for both loads suggests that the wear mechanism during the test is the same.

In the case of nitrided samples, its weight loss was approximately 25 % of the one observed for the non nitrided samples; it was observed also a different rate of weight loss between the samples tested at 50 kg and 125 kg load, but no difference was observed between the top and bottom samples.

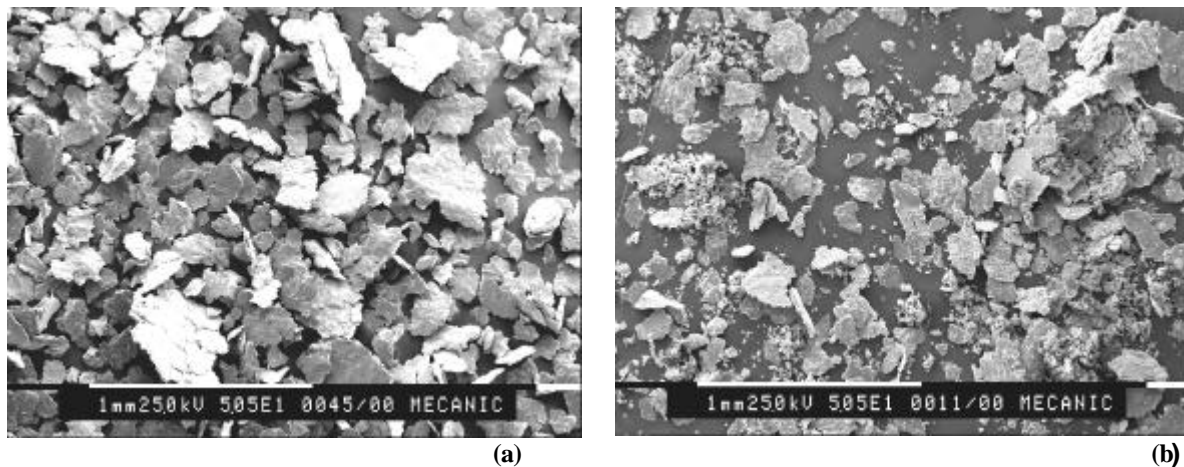


**Fig. 5:** Weight loss of nitrided samples as a function of revolutions.

The wear debris from the non nitrided samples, observed by OM and SEM, does not show any traces of oxidation, it looks bright metallic. The shape of the particles is lamellar of different sizes, with an average size increasing with the number of revolutions and with the normal load. The tests made at 200 kg were interrupted after a number of revolutions lower than for the case of 50 and 125 kg, due to a plastic deformation of the samples, so severe that made impossible to continue the test.

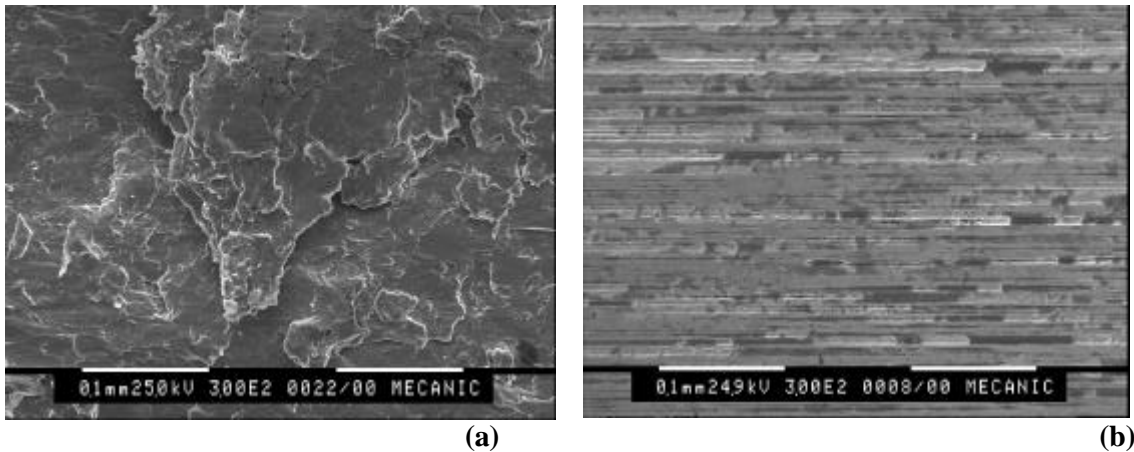
The amount of wear debris for nitrided samples tested at 50 kg load, collected after 17.000 revolutions was very low and its surface seem to be oxidized, while for the test at 125 kg load the wear debris were smaller in size, if compare with the non nitrided samples. According to Sun et. al. (15) a possible explanation for the oxidation of the nitrided surfaces is that, the free energy of formation favours the stability of oxides more than nitrides. In this way, during the wear process, due to the high friction temperature, the nitrogen atoms from the nitrided surface could be replaced by oxygen atoms (16).

Figures 6a and b show scanning electron micrographs of wear debris from samples nitrided and non nitrided respectively tested at 50 kg load, in both cases the shape of the particles is similar (lamellar) which can be associated to the wear mechanism of delamination. It was observed also that the particle size is smaller for the nitrided samples.



**Fig. 6:** Scanning electron micrograph of wear debris obtained after 23.000 revolutions under 50 kg load. (a) non nitrided; (b) nitrided

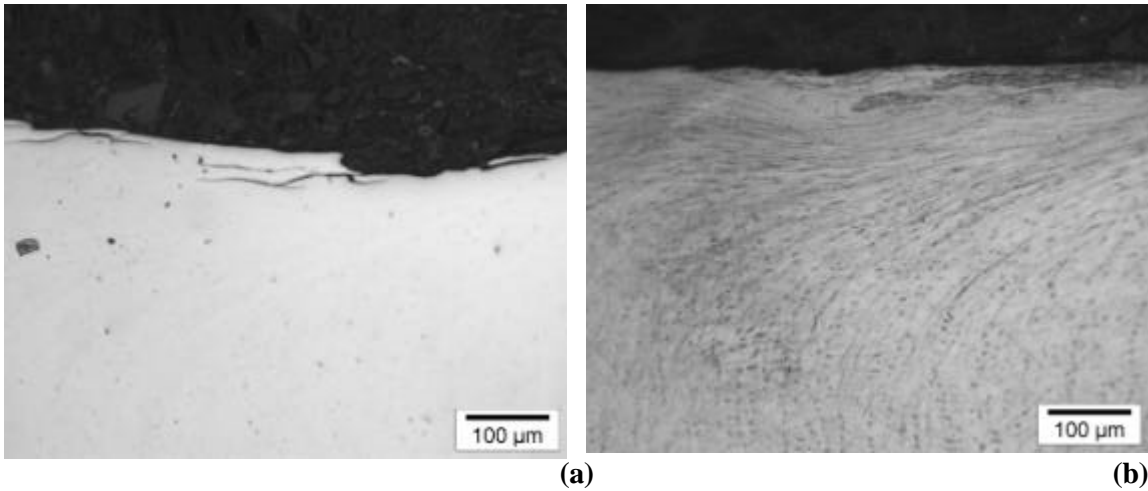
Fig. 7a and b show the surface of the samples non nitrided and nitrided respectively, tested at 50 kg load. In the case of the non nitrided sample, it can be observed that its surface is heavily damage, however its surface appeared bright metallic, similar to the debris. The micrograph of the nitrided sample shows a uniform surface, without any damage at the end of the test (23.000 revolutions). It has been reported (17) that in the case of AISI 316 stainless steel, the friction coefficient increases with nitriding, nevertheless the weight loss is lower with respect to the non nitrided samples, the authors point out that the nitrided layer could reduce the adhesion between the surfaces in contact. The industrial practice recommends to polish the surface after nitriding, in order to reduce the micro roughness generated by the sputtering and nitriding process.



**Fig. 7:** Scanning electron micrograph of surface sample after 23.000 revolutions under 50 kg load. (a) non nitrided; (b) nitrided

Transverse and longitudinal sections of samples tested at 50 kg loads were observed to analyse the subsurface; it was found plastic deformation in all samples but in the ion nitrided. The lack of deformation could be associated to the high hardness of the nitrided layer which inhibits its formation.

The Fig. 8a shows a longitudinal section of a nitrided sample tested at 125 kg load; it can be seen the subsurface flaws parallel to the surface, produced by the wear mechanism of delamination (18). Micro flaws and voids are produced in the subsurface by fatigue; these defects propagate parallel to the surface (19). When the micro flaws find the surface, they generate the particles which become the wear debris. The thickness and shape of the particles depend on the material and test conditions. In the case of Fig. 8b, using Marble reagent, it can be observed plastic deformation in the subsurface, which is consistent with the microhardness profiles obtained in the transverse sections of the samples.

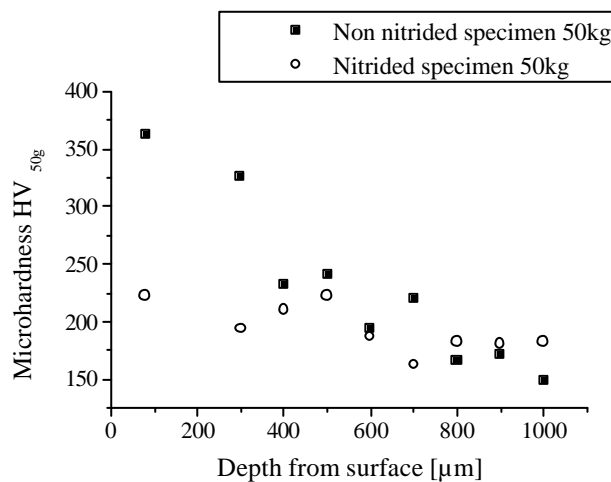


**Fig. 8:** OM of a longitudinal section of a sample tested under 125 kg applied load.  
 (a) Subsurface flaw (b) Subsurface plastic deformation

When a body is under stress, its response to the applied load depends on the load value and also on the material properties. Considering this point, it is possible to estimate the tensile (yield) strength from the hardness data, assuming that the relationship  $Y = HV/B$  holds, where  $Y$  stands for the yield strength,  $HV$  for the hardness and  $B$  is a constant characteristic of the kind of material (19); in the same way the shear yield strength can be assumed as  $K = Y/2$ . When the wear samples were under plastic deformation, the microhardness profile also gives an idea of the work hardening and, consequently, its increase in resistance to farther wear.

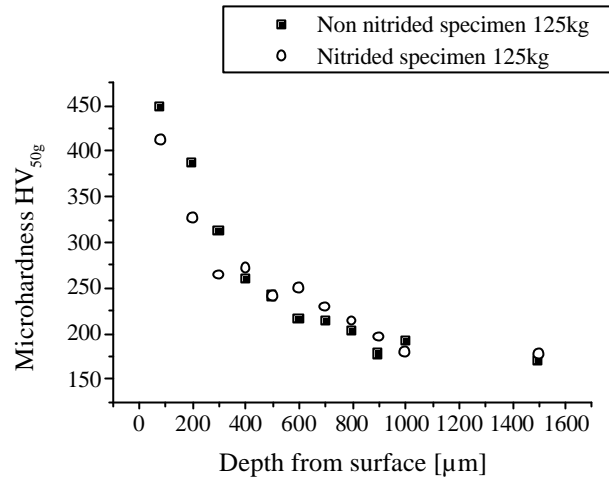
Figures 9a and 9b show microhardness profiles from samples tested at 50 and 125 kg load, nitrided and as received, respectively. The profiles of as received samples, show that they have suffered a big plastic deformation and, considering the equation presented before, we can assume that the shear stress will increase with the normal load applied during the test. In addition, it can be assumed that the shear stress applied to the samples, reaches a value greater than their shear strength, which could explain the plastic deformation and the elimination of the nitrided layer observed on these samples. Conversely, the nitrided samples tested at 50 kg load shows a hardness profile with lower values near the surface, which indicates lower plastic deformation compared to other samples.

In all cases the hardness profiles of the top and bottom samples were similar.



(a)





(b)

**Fig. 9:** Hardness profiles of samples nitrided and non nitrided, tested under 50 (a) and (b) 125 kg load.

According to Bell et. al. (19), the yield strength of the nitrided layer can be established from hardness data, on the other hand it can be evaluated the stress at which the layer it is subject; from this, the load bearing capacity of the nitrides surface can be established. In our case, using the same kind of analysis it is possible to say, from the microhardness profiles and from the micrographs that show the plastic deformation zone, that the load bearing capacity is between 50 and 125 kg load. Below this minimum, the layer supports the load and for values higher than 125 kg it gets destroyed. It also could be accepted that the compressive residual stresses associated to the nitrided layer, would retard the formation of the flows.

## CONCLUSIONS

Ion nitriding improve the wear resistance of austenitic stainless steel type AISI 316L. The nitrided layer has hardness around 1200 HV, which tends to reduce the subsurface plastic deformation on a rolling-sliding test at 50 kg normal load. The nitrided layer appears uniform and formed by two sub layers; a systematic and detailed study of structure and chemical composition needs to be done in order to fully understand the formation of these layers. The principal wear mechanism in the nitrided and non nitrided samples appears to be delamination. In the nitrided samples wear tested at 50 kg load, the mechanism of oxidation also was effective. In all cases the weight loss increased with the load and the number of revolutions. The applied contact stress produced a significant plastic deformation on the surface and subsurface, except on the nitrided samples tested at 50 kg load. Such deformation, observed through the microhardness profiles and micrographs show that at this load level, the stresses at the nitrided layer are lower than its yield strength because at the end of the test the layer still remains.

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