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Mechanical and Microstructural Characterization of AISI 316L Stainless Steel Superficially Modified by Solid Nitriding Technique

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Abstract: AISI 316L austenitic stainless steel superficially modified by the solid nitriding technique was investigated at different nitriding times (2, 4, 6, 8, 12 and 24 h) and at 450 °C. The microstructural characterization was conducted using scanning electron microscopy (SEM) analysis and X-ray diffraction analyses, finding the presence of Fe₂₋₃N, Fe₄N and Cr₂N, among others. The mechanical behavior of the modified surfaces was carried out by developing hardness profiles and relating it with the nitride layer thickness evaluated using scanning electron microscopy (SEM), obtaining layers up to 70 µm wide. The nitrogen diffusion produced species above and below the surface sample with a transformation from the austenitic phase to an expanded austenite (γ_N) phase, which is responsible for producing an increase in hardness of up to 1200 HV in the samples treated at 24 h, which is four times higher than the untreated steels. Wear evaluations of the obtained layers were performed using a pin-on-disk system under zero lubrication, indicating that the samples with 12 and 24 h of treatment present the best wear resistance promoted by an oxidative–adhesive mechanism. The obtained results are positively comparable with those of the ion nitriding technique but with a lower implementation cost.

Keywords: surface modification; stainless steel; hardness; mechanical properties; wear



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1. Introduction

Hardened surfaces of components exposed to a relative friction is an important issue that for many decades has been treated with the goal of minimizing the effect of wear in different areas of the productive sector due to the great economic losses derived from this phenomenon. Therefore, implementing alternatives to harden only the surface of the components that are in contact is important, mainly to reduce their repositioning time, which is one of the imperative goals of this surface engineering [1,2]. On the other hand, obtaining hardened surfaces and a core with a lower hardness is very important, since, in this way, the component toughness may increase, reducing crack nucleation in the hardened zone and, therefore, minimizing the component brittleness [2,3]. Several studies have been conducted to establish that there are many surface treatments available to obtain hardened surfaces. Lin [4] established the importance of the surface texture after nitriding to improve the tribological performance of an AISI 316L. However, there are few methods that involve the combination of two hardening mechanisms such as precipitation and phase transformation [5,6]. Within these methods, nitriding is one of the most important, due to multiple benefits such as uniform surface appearance, very low dimensional variation, controlled depth of the layer and low energy consumption for heating [7–9]. Regarding the ion nitriding technique, Christiansen et al. suggests a pretreatment to prevent a surface re-passivation [1]. Surface modification of stainless steels has been investigated for many years, providing important information about the nitrided layer formation using the ion

nitriding technique [10–14]. It is well known that the main cause of hardness increment in nitrided austenitic steels is produced due to the (γ_N) phase formation [15] and the possible carbonitrides precipitated during the process [16,17] on the surface and in the subsurface due to the nitrogen atom diffusion and the subsequent species formation. However, this technique is expensive due to the high cost of infrastructure; therefore, the implementation of an effective and economical technique for nitride parts is attractive. This is the case in solid nitriding, which consists of diffusing nitrogen atoms into the steel using Fe_4KCN , with the samples being surrounded by this compound and confined inside of a sealed container heated to a certain temperature [18]. Until now, the use of the abovementioned solid nitriding technique remains with limited information [5,18]. Hence, the aim of this present investigation is to generate knowledge of the nitrogen diffusion mechanism and also the mechanisms that improve the hardness and wear behavior of the modified surfaces by solid nitriding of AISI 316L stainless steel. This investigation is carried out at relatively low temperature because if an austenitic stainless steel, such as AISI 316L, is treated at temperatures higher than 450 °C, it can suffer a significant decrease in its corrosion resistance due to the excessive formation of chromium nitride and therefore diminishing the chromium in its solid solution [19]. This work provides an understanding that solid nitriding, through the formation of nitrides of the alloying elements of the steel, can be an effective, reproducible and economical surface treatment to obtain hardened layers in components for industrial applications, which is highly comparable to the ion nitriding technique.

2. Materials and Methods

2.1. Materials Synthesis

Several specimens of AISI 316L-type stainless steel were obtained by cutting coupons with dimensions of $1 \times 1 \times 1 \text{ cm}^3$ (see Table 1 for chemical compositions). Before the nitriding treatments, the samples were superficially pretreated with a based HNO_3 , HCl and HF solution for 3 min. For solid nitriding, the samples were surrounded (20 mm thickness) with a potassium ferrocyanide (Fe_4KCN) compound inside a hermetic stainless steel container (to keep the produced gases inside of the container) internally coated with ammonia nitrate solution (NH_4NO_3) in order to enhance the nitrogen diffusion. After that, the container was placed inside a chamber using a Felisa 301 Series furnace (Guadalajara, Mexico) and heated at 450 °C for 2, 4, 6, 8, 12 and 24 h. Once the nitriding time was over, the containers were removed from the furnace for cooling to avoid the annealing of the samples.

Table 1. Chemical composition for stainless steel AISI 316L in [wt%].

C	Si	Mn	Cr	Ni	Mo	Fe
0.08	1.0	2.0	18.0	11.50	2.30	Bal.

2.2. Metallographic Evaluation

Specimens were sanded using SiC sanding paper up to 600 grade, and afterward they were polished using alumina powder with a particle size of 0.03 μm . The samples were electro-etched with a reactant of 10 g oxalic acid in 90 mL deionized water for 20 s using 0.5 volts in DC for 120 s. Microstructural and surface analyses were carried out in an LEO-1450 VP Scanning Electron Microscope (Southampton, UK), whereas chemical microanalysis was performed using the energy disperse system (EDS) attached to the equipment. X-ray diffraction analyses were carried out using a Bruker D2 PHASER X-ray diffractometer equipment (Billerica, MA, USA), with a radiation of $\text{Cu-K}\alpha$: $\lambda = 0.15406 \text{ nm}$ and a $3^\circ/\text{min}$ scanning speed.

2.3. Mechanical Testing

Hardness Vickers measurements were carried out on the sample surface (previously polished with diamond paste with an average size $0.03\text{ }\mu\text{m}$), using an LECO, LM300AT Microhardness Tester (St. Joseph, MI, USA), with a 0.2 kg load for 15 s . For this, a series of 10 indentations in different zones of the surface sample were taken to obtain a better precision in the average and standard deviation of the hardness value, which is in agreement with ASTM E384-22. Five samples of each time of treatment were used. A conventional pin-on-disk TRB³ Anton Paar Tribometer (Graz, Austria) wear system was used to evaluate the wear behavior of the pin samples with dimensions of 6 mm diameter by 8 mm length and a surface roughness with alumina $0.5\text{ }\mu\text{m}$, under a load of 5 N , with a constant disk rotation of 100 rpm and zero lubrication. AISI-4140 steel (oil quenched) was used as a counterpart with a hardness of $51 \pm 3\text{ HRC}$ in agreement with ASTM-G99. Worn surfaces were observed and analyzed using a SEM (scanning electron microscopy, Leo-1450 VP, Southampton, UK) and a total of three samples were used to calculate wear factor, weight loss and worn surfaces analyses. A Mitutoyo SurfTest SJ-210 JP Profilometer Roughness Tester JP (Kawasaki, Japan) surface profilometer was used to determine the surface roughness before and after solid nitriding. The surface roughness was characterized by measuring the average roughness, R_a , according to ISO 21920-2:2021. The measurement length was chosen as 0.5 mm . For each specimen, the surface roughness was determined developing 10 measurements, and the results are shown in Table 2.

Table 2. Roughness values for the different nitriding times before and after treatment.

Treatment Time [h]	Surface Roughness Before Solid Nitriding [μm]	Surface Roughness After Solid Nitriding [μm]
2	0.030	0.113
4	0.032	0.127
6	0.027	0.145
8	0.031	0.157
12	0.027	0.194
24	0.026	0.216

3. Results and Discussion

3.1. SEM Characterization

Nitrogen Diffusion and Line Scan Characterization

Diffusion in steel is considered as a mass transport process in a non-steady state where the atoms that diffuse into the steel do so as a function of time, concentration and temperature in the system [15,16]. The formation of a primary and secondary layer is produced due to the initial nitrogen diffusion reaching a saturation point, and then, a secondary layer starts to be created with the nitrogen atoms that diffuse from the primary layer [17,19–21]. Analyses were carried out on the cross section of the treated samples, with the acronym used in the figures being as follows: P for the primary layer, S for the secondary layer and DL for the diffusion layer. Figure 1A shows the image of the sample treated for 2 h . The presence of a primary layer of approximately $6\text{ }\mu\text{m}$ wide can be observed, with a secondary layer of approximately $2\text{ }\mu\text{m}$, with the total depth of the diffused layer being $35\text{ }\mu\text{m}$ with a nitrogen surface concentration of $5.28\text{ at.}\%$. Similar behavior was observed in Figure 1B for the sample after 4 h , which presents a nitrogen surface value of $7.8\text{ at.}\%$. For the treatment for 6 and 8 h , shown as a primary layer of approximately $17\text{ }\mu\text{m}$ with a secondary layer of $6\text{ }\mu\text{m}$, as seen in Figure 1C,D, the layer is observed as homogeneous. Although, it is interesting to observe the apparition of several precipitates along the nitrided layer segregated mainly to the interphase between the primary and the secondary layer across the modified layer, which is produced due to the reaction between the nitrogen with the constituent elements of the steel [22–26], with the total depth of the layer being $50\text{ }\mu\text{m}$. For the treatment for 12 and 24 h in Figure 1E and 1F, respectively, it is observed that the

primary layer formation of 19 μm with a secondary layer of 8 μm approximately has few precipitates with an average size of 0.3 μm . These are the largest diffusion layers obtained in these experiments with a maximum width of 70 μm on average and a surface nitrogen content of 9.40 at.%.

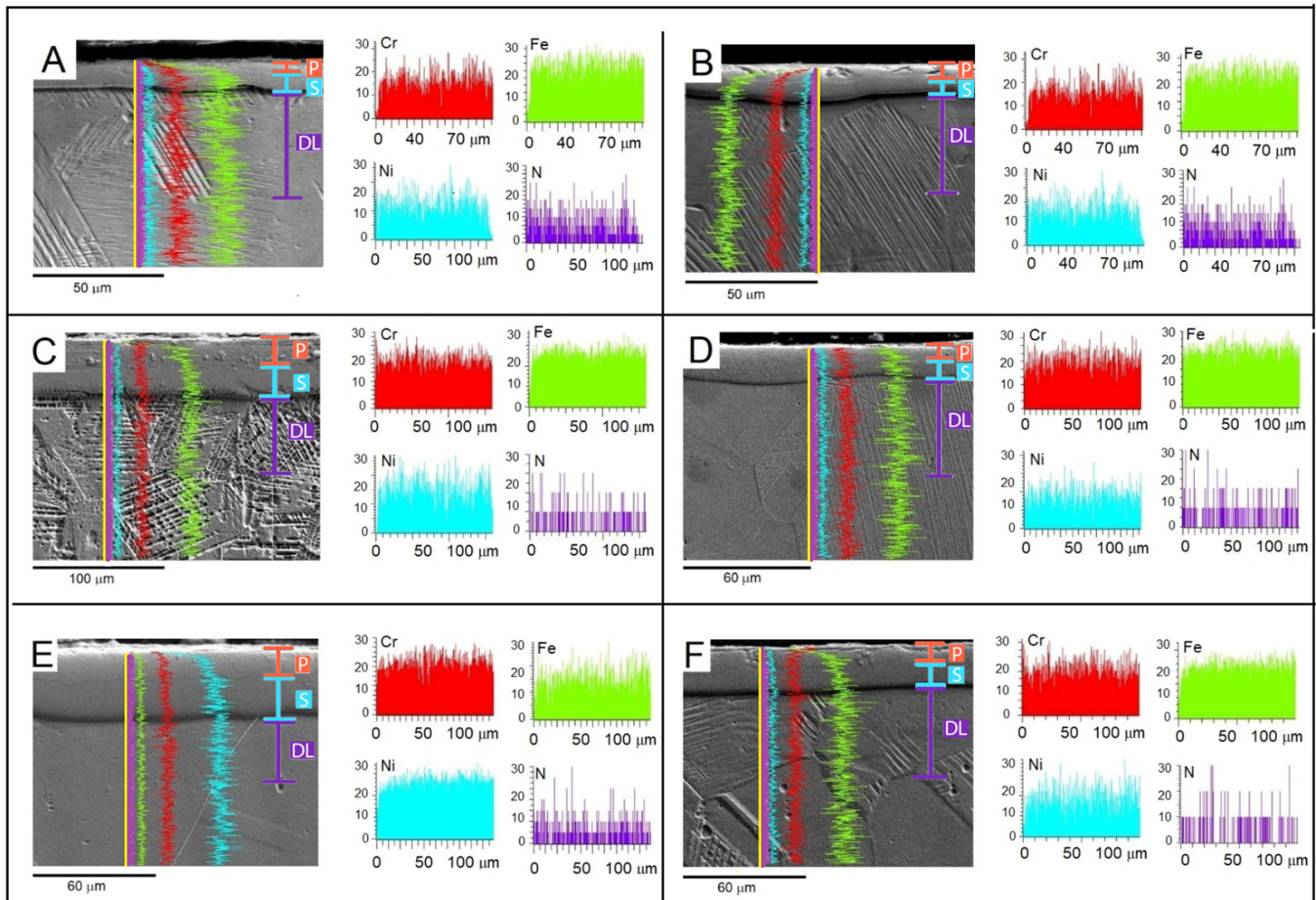


Figure 1. Nitriding layers morphologies at different treatment times: (A) 2 h, (B) 4 h, (C) 6 h, (D) 8 h, (E) 12 h and (F) 24 h. The line scan profiles show the nitrogen diffusion toward the matrix sample. Total diffusion layers cannot be clearly observed in the images of Figure 1 due to the over-etching of the samples.

Nitrogen chemical analyses are observed in the nitrogen diffusion profiles of Figure 2, which were obtained by performing punctual chemical analyzes along the nitrided layer from the surface to the center of the treated sample, supporting the results observed in the line scan analyses. It can be observed that the samples with 2 and 4 h of treatment present 34 and 39 μm of diffusion, respectively, while the samples with 6, 8, 12 and 24 h of treatment show a nitrogen diffusion of 55, 60, 66 and 70 μm , respectively. It is interesting to observe that there is an interval between the sample with 4 h and the sample with 6 h of treatment of approximately 15 μm of nitrogen diffusion. This effect can be attributed due to the strong nitrogen concentration on the surface sample with 6 h of treatment that again promotes the continuous nitrogen diffusion. In other words, nitrogen atoms can move from one position in the lattice to another, through an activated process in which the ion moves over an energy barrier [5].

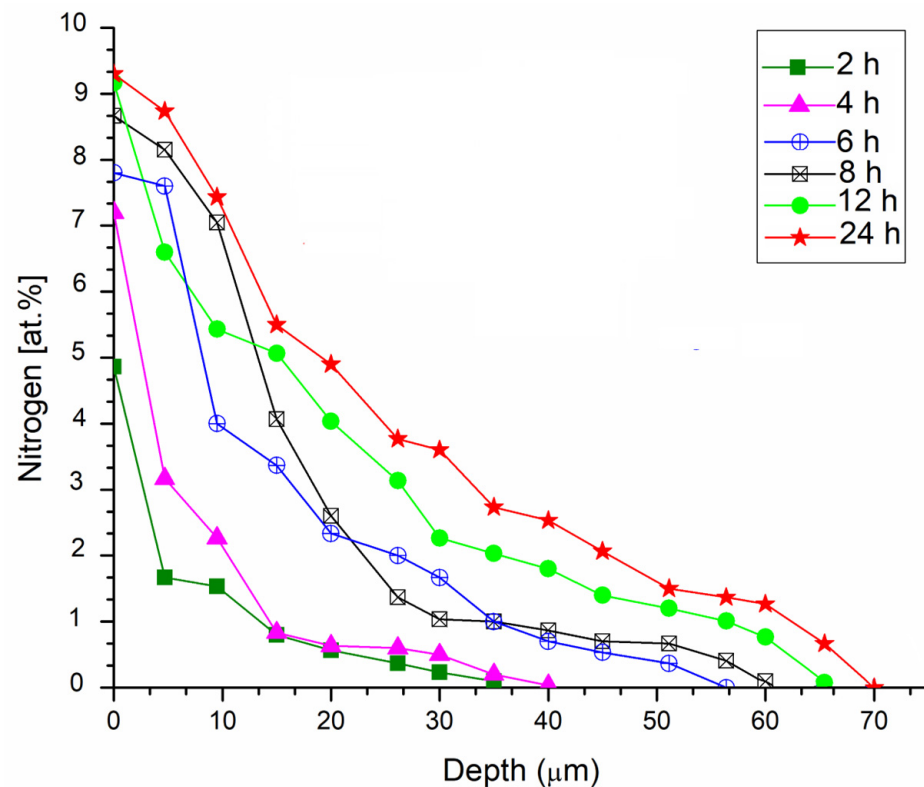


Figure 2. Nitrogen diffusion profiles obtained after the solid nitriding process with treatments of 2, 4, 6, 8, 12 and 24 h.

3.2. X-ray Diffraction Analyses

Figure 3 shows the XRD patterns of the solid nitriding samples treated with different exposure times. Due to the atomic radii of nitrogen being 0.5427 Å, the diffusion mechanism is clearly interstitial [27], where nitrogen diffuses into the sample and reacts with the γ -phase (matrix) and the other elements of the steel (Fe, Cr, Ni) [28–32]. Therefore, the formation of the γ_N phase is indicative that the nitriding process has been obtained [17]. For the sample treated for 2 h, the XRD spectrum mainly shows the presence of γ_N and γ' -Fe₄N formed during the treatment. The presence of Fe- α and Cr- α was detected in several samples with different times of the solid nitriding treatment. In the sample treated for 4 h, it mainly shows the presence of γ' -Fe₄N, while the CrN phase does not appear in this diffraction pattern (and neither in the others patterns), which is indicative that chromium does not react completely with nitrogen, creating chromium nitrides, which is one of the main objectives of not affecting the corrosion resistance of the steel [19]. For the treatments for 6 and 8 h, the formation of Cr₂N, ϵ -Fe₂N and ϵ -Fe₃N was detected, together with the α -Fe presence, while in the diffraction pattern for the samples treated at 12 and 24 h, the presence of Cr₂N and Fe₄N can be observed. The formation of these nitrides for both nitriding times may indicate that the nitrogen, which comes from the potassium ferrocyanide, has been depleted throughout the treatment due to temperature, i.e., the nitrogen concentration has decreased at very low levels, reducing the possibility of a continuing diffusion. The ICDD files used to identify the peaks were Fe₄N-(111), ICDD: 98-0030-05401; Fe₂₋₃ N-(110), ICDD: 98-009-8404; Fe- α -(110), ICDD: 98-007-1819; Cr₂N-(110), ICDD: 98-002-5566; and Cr- α -(200), ICDD: 01-085-1336.

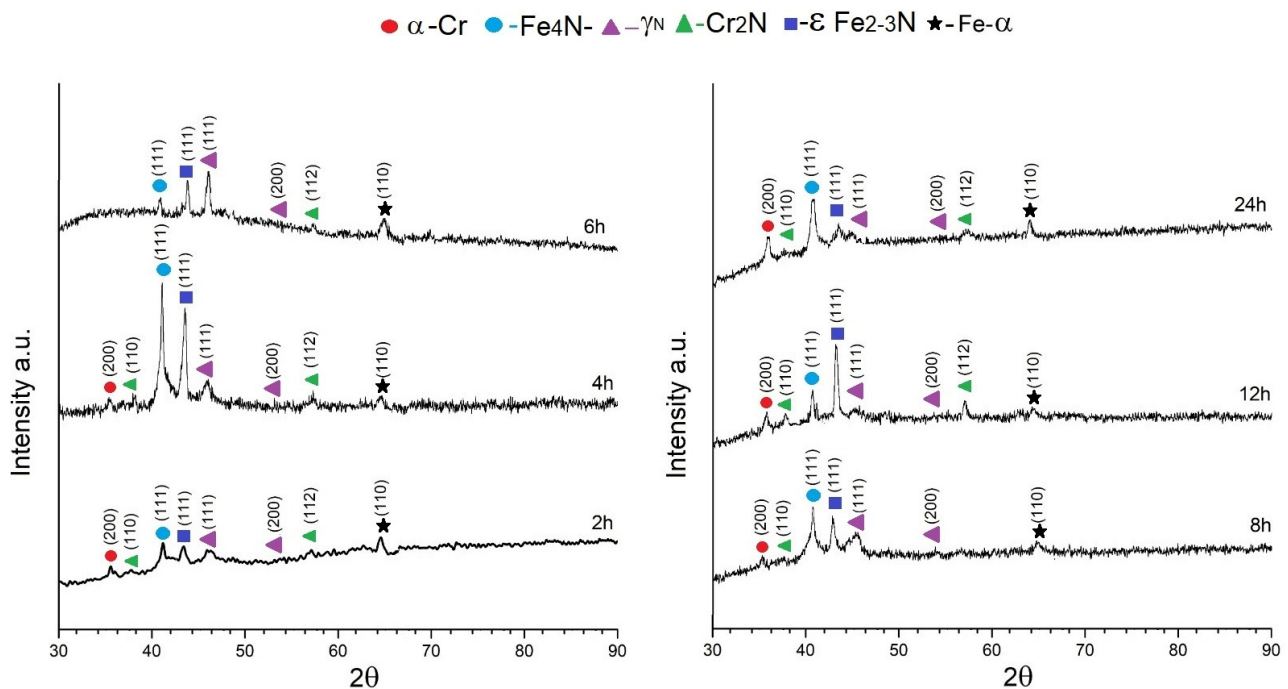


Figure 3. X-ray diffraction patterns of AISI 316L stainless steel with solid nitriding treatment with times of 2, 4, 6, 8, 12 and 24 h.

3.3. Mechanical Properties

3.3.1. Microhardness Evaluation

Hardening after the nitriding process is mainly attributed to the (γ_N) formation [15,16] in addition to the formation of different types of nitrides such as Fe_2N , Fe_4N and Cr_2N [18]. Figure 4 shows the hardness values for the solid nitriding samples, and also shows the hardness value for the untreated sample of 280 HV as a reference. Hardness values were obtained along the cross section of the treated samples from the surface sample to the final sample of the modified layer. For the treatment time of 2 h, an average hardness of 1010 HV with a depth of 48 μm occurs, showing a sudden decrease in hardness after 5 μm , perhaps after the primary layer finishes due to the compounds formation, which is basically due to the γ_N phase formation [16]. On the other hand, it is important to mention that hardening in the modified layer cannot be exclusively attributed to the formation of the nitriding compounds, because it is also due to the lattice distortion produced by the diffusion of nitrogen within the austenitic structure via the interstitial mechanism [18,31]. The sample with 4 h of treatment had a hardness value of 1104 HV with a hardened layer of 45 μm , also showing a drop in hardness of approximately 6 μm . For the sample treated for 6 h, a hardness of 1116 HV was obtained over the surface with a hardened layer of 80 μm . In this case, the drop in hardness occurs at 25 μm below the surface. These hardness drops observed in the plots can be correlated with the change in layer morphologies observed in Figure 1, from the primary layer to the secondary layer, i.e., the zone where there is a transition of compound formation from high saturation to a constant nitrogen diffusion [9,27]. That is, if the concentration of nitrogen atoms is lower than the diffusion rate of nitrogen driven by the atoms gradient at greater depths, a layered structure of Fe-N phases (interface) can develop at different layer depths, which is a function of the decrease in the chemical potential of nitrogen as the distance from the surface increases. Hence if the compositions on both sides of this interface are equal to the compositions prevailing under equilibrium conditions, then a local equilibrium occurs. However, if there is an accumulation or decrease in nitrogen atoms on either side of the interface, then the local equilibrium is affected because the transport of nitrogen atoms across the interface is not restricted, generating a rapid mobility of nitrogen atoms through this interface

and, therefore, a real equilibrium can be reached again, according to the applied nitriding temperatures [27,31]. For the 8 h treatment, a hardness of 1090 HV was obtained with a depth of 60 μm . For the 12 h sample, a hardness of 1106 HV was obtained with a depth of 90 μm . Finally, in the case of the sample treated for 24 h, a hardness of 1200 HV was obtained with a depth of 65 μm . Thus, the hardness increment reached by this thermochemical treatment was approximately five times higher in comparison with the untreated sample, attributing this hardening behavior from the microstructural point of view to the creation of defects such as precipitates and dislocations due to lattice distortion [32] in the structure that inhibits the plastic flow [7,9,23]. The hardness reached by this treatment is comparable with the results in hardness reported by Lin et al. [4] of 1050 HV in plasma-nitrided AISI-316 stainless steel and the results reported by Gokcekaya et al. [15] in a range of 900 to 1400 HV in ion-nitrided AISI-316. The standard deviation in the hardness measurements was of 0.5% on average, which indicates a variation of 5.5 HVN.

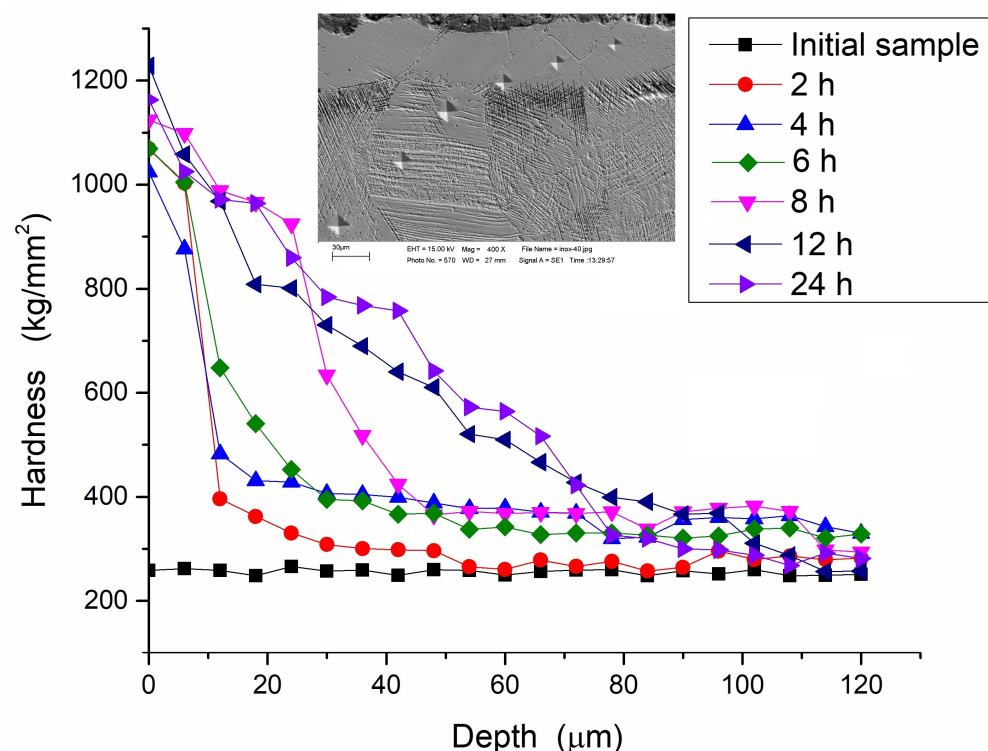


Figure 4. Hardness values for the samples with different diffusion treatments compared with the untreated sample. The inset image shows the dimensions and distribution of the indentations in the 24 h sample.

3.3.2. Wear Behavior

Weight Loss Analyses

The weight loss curves as a function of sliding distance are shown in Figure 5. In this figure, the wear behavior of the samples is observed with solid nitriding treatments for 2, 4, 6, 8, 12 and 24 h compared with the sample without treatment (initial sample). It can be observed that the initial wear behavior of the sample after 2 h treatment in a range from 0 to 450 m increases slightly. After this distance, the weight losses present a gradual increment until the end of the test. This sudden increment is due to the coupling between the two surfaces during the wear process. In the case of the samples treated with 4, 6 and 8 h, the initial wear remains without increasing until 700 m. After this distance, it increases up to 1000 m to form a plateau until 2000 m, and finally there is a slight increase in the weight losses until the end of the test. This behavior can be attributed to the intrinsic superficial hardness of the samples reached by the formation of the γ_N phase [4,15,27]. For the samples treated with 12 and 24 h solid nitriding treatment, a remarkable wear resistance is observed

from the initial to the end of the test at 4000 m without any apparent weight loss in these samples. The moderated changes in the curves indicate the formation of a stable oxide interface adhered to the worn surfaces. This oxide layer is created by the relative frictional heating that produces a strong bond between the formed layers due to the high hardness of the steel surface [4].

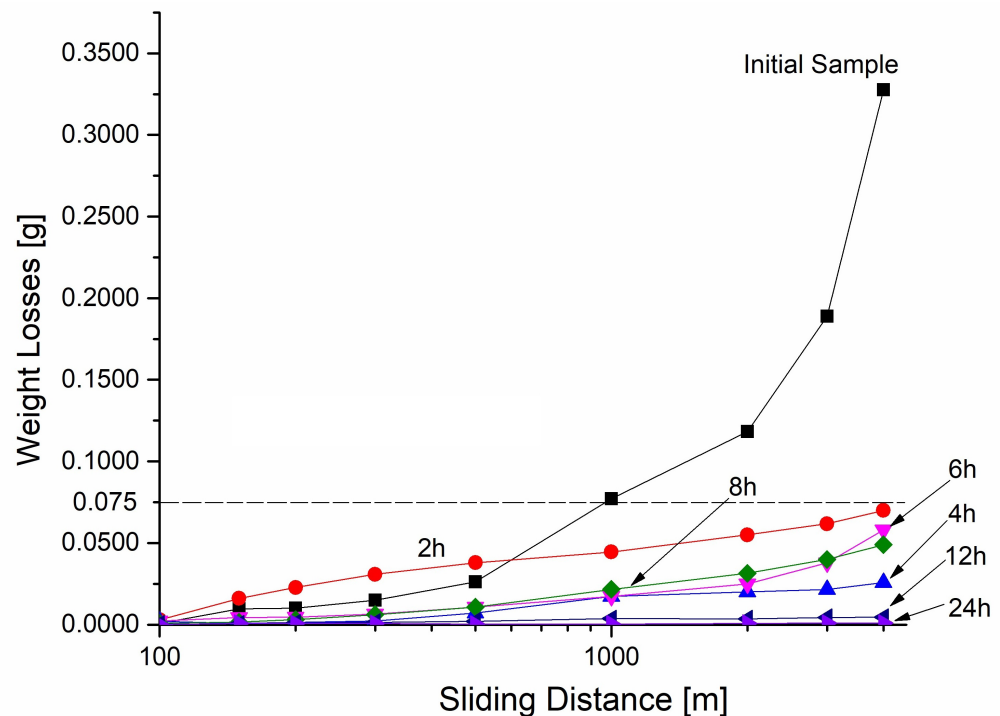


Figure 5. Weight losses against sliding distance of the samples with solid nitriding treatment: initial sample, 2, 4, 6, 8, 12 and 24 h.

Worn Surfaces Analyses

The analysis of the worn surface is presented in Figure 6, where it can be observed that for the initial sample (untreated sample) in Figure 6A, abrasion grooves are observed in the sliding direction with small zones with particles adhesion, which is characteristic of the abrasion mechanism [3,13]. The elemental analysis obtained shows the presence of oxygen in small quantities in addition to Cr, Fe and Ni in a greater proportion, suggesting that there is no significant oxide formation with the elements present in the steel. For the treatment for 2 h in Figure 6B, deep grooves over the wear area are mainly observed, where it clearly shows the sliding direction and presents the accumulated material with small adhered particles. The chemical analysis in this area shows the presence of oxygen in a greater proportion as well as a high percentage of Cr, Fe, Ni. These elements are part of the worn material, although this finding does not exclude the possibility that Fe comes from the counterpart [29]. In Figures 6C, 6D and 6E, which correspond to the worn surfaces of the samples with 4, 6 and 8 h of heat treatment, respectively, a pattern of few grooves is observed on the worn surface with the presence of oxide accumulation, which implies that there is an oxygen reaction with the elements present in the surface sample as is shown in the chemical analyses of the worn surfaces where the presence of oxygen is high. On the other hand, the images of the worn surfaces corresponding to the samples with 12 and 24 h of treatment are presented in Figure 6F,G where few zones with oxide agglomeration are observed (indicated by an arrow), although grooves are observed in most of the area, but with a low level of depth. In the chemical analyses of these samples, a relatively low concentration of oxygen was detected along with Ni, Cr, Fe and Mo. These results broadly support the observations of the wear curves, thus establishing that the operating wear mechanism is abrasive with a mixed oxidative–adhesive mode [3,13,29]. On the other hand,

Mo was not detected in several samples due to the presence of dense oxide layers formed on the surface sample after wear, which mask the molybdenum presence and therefore inhibit the Mo detection in these specific zones.

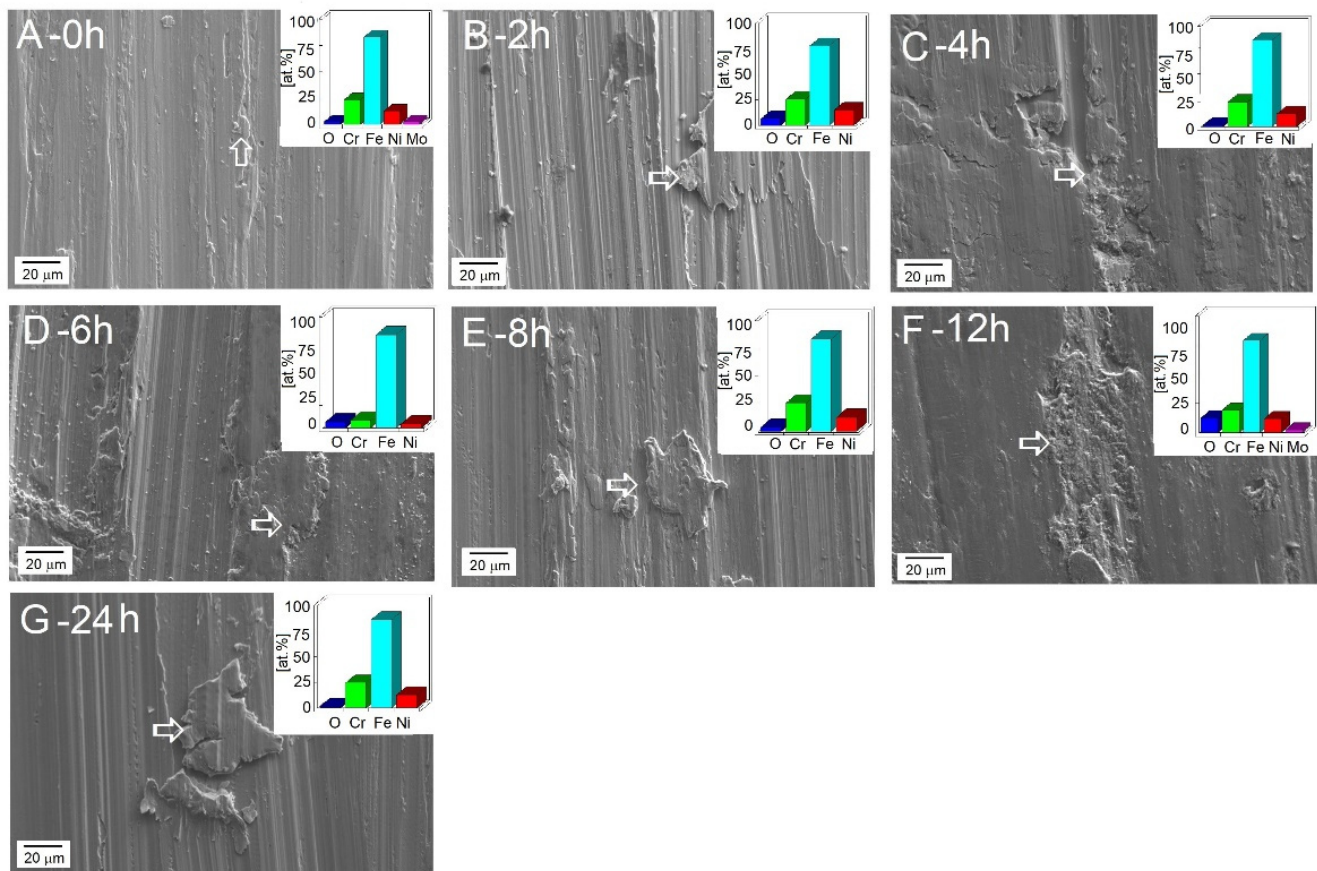


Figure 6. Worn surfaces images of the samples with solid nitriding treatment: (A) initial sample, (B) 2 h, (C) 4 h, (D) 6 h, (E) 8 h, (F) 12 h and (G) 24 h.

Wear Factor Analyses

Because a wear phenomenon involves the weight loss as a function of time and the sliding distance, then it is important to know the wear index between these parameters, hence the results of this indicator is presented in Figure 7 where the graph of the wear factor obtained from the wear curves of the initial sample and 2, 4, 6, 8, 12 and 24 h of treatment is presented. In the graph, a noticeable value of the wear factor for the initial sample is observed, which is approximately 80% higher compared with the treated samples. This phenomenon is generated by the difference in hardness between the pin sample and the counterpart [29] and is notoriously influenced by the amount of Cr and Fe nitrides generated on the worn surface [13,33] and the oxides of the elements present in the steel. For the worn samples treated during 2, 4, 6 and 8 h, a resultant wear factor average of 1.2×10^{-5} g/m was obtained while for the worn samples treated for 12 and 24 h, a wear factor of 5×10^{-6} g/m was obtained. These results clearly indicate the advantage of implementing a treatment for 12 and 24 h, where a minimum value in the wear factor is achieved and consequently an increase in the useful life period of the steel [13,29]. The measured error in the experiments was of 1.8%, which falls within the experimental error.

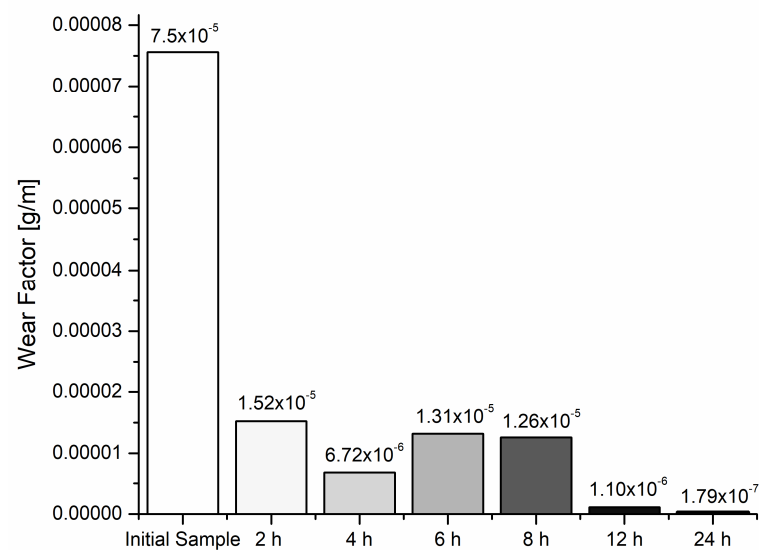


Figure 7. Wear factor of the samples with solid nitriding treatment compared with the untreated sample. Treatment times of 2, 4, 6, 8, 12 and 24 h.

4. Conclusions

In this research, the scientific and technological information related to the microstructural and mechanical behavior of an AISI 316L stainless steel surface modified by the solid nitriding technique at 450 °C and different nitriding times is presented. The following conclusions were obtained:

1. The layer thickness consisting of different nitrides increases as a function of the nitriding time.
2. The γ_N , γ' -Fe₄N, Cr₂N and Fe₂₋₃N phases were produced in the diffusion layer in most of the different nitriding times. Although at 12 and 24 h, the γ_N phase decreases due to the reduction in nitrogen concentration.
3. The samples treated for 24 h exhibited the highest surface hardness values. This hardness increment is attributed to the presence of the γ_N phase, which is one of the main factors responsible for the hardness increase, followed by the lattice distortion of an interstitial mechanism.
4. Wear evaluation indicates an increase in wear resistance in the samples treated at 12 and 24 h exposure times, clearly evidenced and supported by the wear factor results, with the different nitrided phases being responsible for this increase in wear resistance, and the produced oxide layers due to intrinsic frictional heating. Hence, they suffered a small depletion due to the low residual stresses in the nitrided layer.
5. A disadvantage of applying this method is that the pieces obtained have a rough surface finish after the solid nitriding treatment. However, with a polishing treatment, an acceptable surface finish is obtained without affecting the modified layer.

The nitrided sample at 24 h exhibited a thick nitrided layer with minimal or zero cracks and/or delamination generation, with the best wear and hardness performance. This sample is suitable for use in wear and corrosion applications. Therefore, by applying this method under these conditions, results similar to those of ion nitriding can be obtained.

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