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Plasma nitriding of AISI 431 steel: surface hardness - hardness profile [Nitruración por plasma del acero AISI 431: dureza superficial – perfil de durezas]

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Resumen

El presente trabajo investigó, el efecto del tiempo de nitruración en plasma en el rango de 5 a 15 horas, sobre el perfil de durezas de la sección transversal de muestras de acero inoxidable AISI 431; además de tomar y diferenciar los datos de la dureza superficial, profundidad de capa efectiva y espesor de capa de nitruros. El proceso de nitruración fue por plasma, la temperatura del proceso se mantuvo constante en 400 °C. Las muestras evaluadas fueron maquinadas (cilindradas y refrendadas), y se dejaron en un diámetro de una pulgada y una pulgada de longitud. Los tiempos de 10 y 15 horas de tiempo de nitruración se obtuvieron por acumulación de tiempo de 05 horas de nitruración por semana; los perfiles de dureza se obtuvieron mediante el uso del micro durómetro marca LECO modelo LMV-50V; la norma ASTM E3-91 fue utilizada para la toma de los datos de dureza mencionados, de estos se pudo determinar que las durezas superficiales máximas son (1053, 1252 y 1327) $HV_{-0.01}$, para los tiempos de nitruración de (5,10 y 15) horas respectivamente, los espesores de capa efectiva promedio fueron de (37.75, 33 y 28.75) μm; mientras que los espesores de capa de nitruros fueron de (4.9, 7.03 y 10.7) μm correspondientes a tiempos de (5, 10 y 15) horas respectivamente. La dureza en el núcleo después del tratamiento de nitruración se mantuvo en el rango de (275-277) $HV_{-0.01}$. Estos valores se determinaron mediante la evaluación microscópica de las muestras ensayadas, el reactivo para metalografía utilizado fue Nital al 3% mediante ataque electrolítico por 3 minutos en cada caso. El análisis estadístico correspondió a pruebas “t” de student, en la forma de comparación por pares, de la cual se determinó, la no diferencia significativa entre repeticiones y la diferencia significativa entre los diferentes niveles de estudio.

Palabras clave: AISI 431; Nitruración por plasma, Dureza, Capa de nitruros.

Abstract

The present research evaluated the effect of the nitriding time in plasma in the range of 5 to 15 hours, on the hardness profile of the cross section of stainless steel samples AISI 431; in addition to taking and differentiating the data on surface hardness, effective layer depth and nitride layer thickness. The nitriding process was by plasma, the process temperature was kept constant at 400 °C. The evaluated samples were machined (rolled and countersigned), and were left in one inch diameter and one inch in length. The times of 10 and 15 hours of nitriding time were obtained by accumulating time of 05 hours of nitriding per week; the hardness profiles were obtained by using the LECO model LMV-50V micro durometer; The ASTM E3-91 standard was used to collect the aforementioned hardness data, from these it was possible to determine that the maximum surface hardnesses are (1053, 1252 and 1327) $HV_{-0.01}$, for nitriding times of (5,10 and 15) hours

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respectively, the average effective layer thicknesses were (37.75, 33 and 28.75) µm; while the nitride layer thicknesses were (4.9, 7.03 and 10.7) µm corresponding to times of (5, 10 and 15) hours respectively. The hardness in the core after the nitriding treatment was kept in the range of (275-277) $HV_{0.01}$. These values were determined by microscopic evaluation of the tested samples, the metallography reagent used was 3% Nital by electrolytic attack for 3 minutes in each case. The statistical analysis corresponded to Student's “t” tests, in the form of pairwise comparison, from which the non-significant difference between repetitions and the significant difference between the different levels of study were determined.

Keywords: AISI 431; Plasma nitriding, Hardness, Nitride layer.

1. Introduction

The metal mechanic, automotive and plastic industry makes use of various varieties of steels for the manufacture of its multiple components; Depending on the use to which said component is going to be subjected, the selection of the steel and the subsequent thermal or thermochemical treatment to be applied are made and thus, provide it with the necessary properties to prolong its useful life (Apraiz, 1891). Many heat treatments have been used to improve the mechanical properties of the surfaces of the steels since this area is the one that ends up causing a number of failures in service of these components, such properties that are usually paid attention are the hardness and resistance to wear. One of the thermochemical treatments of great diffusion at an industrial level is nitriding, which is used for metallic materials such as steel, this thermochemical treatment improves the surface mechanical behavior of steels, due to the improvement in surface hardness, resistance to wear. and corrosion, in addition to being easy to apply and reasonable in cost / benefit terms. Among the three different types of nitriding processes, liquid, gas, and plasma, the latter can be considered the most requested, because it presents positive characteristics such as precise control of the surface layers, low working energy and relatively low consumption of gas, does not generate environmental pollution and allows heat treatments at low temperatures (below 500 °C), a safe temperature in the case of stainless steels, since otherwise the formation of chromium carbides is encouraged, thus increasing the possibility awareness of steel. Ion nitriding is a process of thermal-physical-chemical treatment that causes surface hardening by interstitial diffusion of atomic nitrogen on steel surfaces, and the formation mainly of a layer of nitrides called white layer by color in light of an optical microscope (Alves, 2012).

Nitriding can significantly increase the surface hardness and wear resistance of steels, depending on the treatment conditions and the chemical composition of the steel samples; the distortion of nitrided workpieces is also small because nitriding is carried out below the austenitic phase transformation temperature, with only the effect of temperature expansion remaining as a possibility of distortion, but eliminating the phase transformation distortion (Han, 2014). Obtaining an optimal nitrided layer depth, as well as good toughness and hardness properties in nitrided areas, are a function of the temperature and treatment time, in addition to the treatment after the nitriding process; These parameters are important in obtaining the nitrided component with good mechanical properties, both on the surface and in the core (Hey, 1995). In our environment, the plasma nitriding process is relatively new, so plasma treatments appear in the Peruvian market at an industrial level, and as a new process in the market, it generates distrust with respect to its competing treatments such as nitriding processes by gases and / or nitrucarburization processes by liquids, in this way we propose to demonstrate the efficiency of the plasma process, both in costs and in quality of service and delivery time; This is how we define characterizing the variation of the properties of some commercial steels and those of greater demand, these being the surface hardness, effective depth, thickness of the nitride layer, resistance to corrosion, resistance to wear; Among the main steels of greatest commercial interest for nitriding processes, we have AISI 4140, AISI 4340, AISI 1045, AISI 431, AISI D2, AISI H13 among others, therefore there is a need to evaluate the benefits of this treatment in the aforementioned steels ;, within the framework of the aforementioned, the following research work is presented that evaluated the effect of the plasma nitriding treatment of AISI 431 steel, on some of the mechanical properties of greater demand in the national market, the Information will be relevant in companies that consume these steels, such as companies that produce parts with thermoplastics, mold jaws, container sealing jaws, among other elements. Nitriding is carried out under ideal conditions on steels previously quenched and tempered at a temperature higher than that used in nitriding; The pieces must be in the final dimensions, since after nitriding no machining operation should be carried out, only polishing, otherwise the nitride layer formed would be reduced; On the other hand, plasma is considered the fourth state of matter, by applying a sufficient amount of energy to a gas, the ionization phenomenon occurs that

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allows the appearance of electrically charged particles - ions and electrons -, when the particles present a force electromagnetic that defines the system, the gas is said to have been transformed into plasma; The energy used to make the gas react can, in theory, be of any type; be it thermal, mechanical or electrical, the latter is the most used for plasma nitriding. Plasma nitriding is a thermochemical treatment that consists of incorporating N into the surface of the steel by sputtering under reduced pressure (sub-atmospheric); Plasma nitriding is presented as an alternative process to traditional methods in a gaseous atmosphere or in a molten salt bath, and its main advantage is a reduction in the process time and energy consumed as well as a better control of the microstructure of the layer nitrided, managing the parameters of the electric discharge; The process consists of placing the part (s) to be nitrided in a refrigerated chamber (cold-wall furnace type), it is vacuum, then a controlled mixture of gases is introduced, a potential difference is created between the part (cathode (-)) and furnace wall (anode (+)), thus having control over process variables such as temperature, chamber pressure, time, potential difference and atmosphere, the nitrided layer is produced with the aforementioned characteristics (Dean, Graziano , Mortarino, Firrao, 2011). Process temperatures vary from 350 °C to 580 ºC, with pressures from 0.1 to 1kPa, nitriding cycles range from ½ to 10 hours. Thanks to ionic bombardment, atoms of some pollutants are released from the surface of the material; the iron in the material reacts with nitrogen and forms iron nitrides (FeN), which causes the formation of a hard layer known as a “white layer”, the chemical composition of this layer can vary depending on the chemical composition of nitrided steels ; the increase in the temperature of the piece and the ionic bombardment of nitrogen allows the diffusion of nitrogen atoms into the structure of the material, which forms nitrides with the alloying elements of steel such as, for example, chromium nitrides that protects the surface and could provide resistance to wear and corrosion; In addition, it increases the hardness in depth and resistance to fatigue (Cirimello et al., 2018).

Moina et al. (2002), evaluated the effect of ionic nitriding of martensitic stainless steels, concluding that nitriding treatments carried out for 20 h at 400 and 500ºC for AISI 410 martensitic steel produce a hardness greater than 1000 HV on the surface and profiles with abrupt interfaces due to rapid precipitation of CrN on the nitriding front. The maximum hardening effect was obtained in the nitrided samples at 400ºC (1400 HV). The nitrided layer thickness for both treatments was approximately 30 µm and the samples treated at 400ºC presented a higher density of cracks and nitrides preferentially formed at the grain edges. Higher nitriding temperatures result in higher average roughness and size of micro-formations. On the other hand, Charadia (2001) evaluated the nitriding of stainless steels using a pulsed plasma, he concluded that during nitriding for AISI 304 steel, the austenitic structure and the amount of alloys make diffusion difficult and are always found in stainless steels. finer layers of compounds and there is a very small diffusion zone and less hard than in ferritic steels. Regarding the temperature, at less than 410 °C, the surface hardness is not high, as if it were operated at higher temperatures. With four hours of treatment at 430 °C, a 4.7 µm nitrided layer was achieved that raises the micro hardness measured on the surface from (220-240) HV, corresponding to untreated steel, up to (450-470) HV, approximately 20 to 45 HRC, measured directly on the surface. Cimeta and Suarez (2004), evaluated the nitriding of an AISI 410 stainless steel starting from different thermal treatments, they concluded that the ionic nitriding of martensitic steels produces a notable increase in surface hardness and wear resistance; In the cases of starting from a material annealed or quenched and tempered at 600 °C, the specimens lose only 25% of material in the wear test with respect to the standard specimen; in the case of starting from a quench and temper at 400 °C, it results in a 16% decrease in the wear rate. Brühl et al. (2009), evaluated the ionic nitriding of stainless steels hardenable by precipitation, determined that the ionic nitriding of steels hardenable by precipitation, increases the hardness by the formation of a layer rich in nitrogen of between 10 and 20 microns, at values higher than 1000 HV in all cases; This hardness increases its resistance to wear by 35% on average, even taking into account that the abrasion test is relatively aggressive due to the hardness of the sand, which is composed mainly of quartz; It is then demonstrated with this work that ionic nitriding at low temperatures (less than 400 °C) is an effective treatment to improve the tribological properties of steels hardenable by precipitation, without affecting their resistance to corrosion). Brühl et al. (2008) evaluated the ionic nitriding of high-chromium martensitic stainless steels, determined that the nitriding of Boehler's martensitic steels, M333 and M340, increases the hardness due to the formation of a layer rich in nitrogen, but deteriorates the resistance. corrosion, evidenced in a salt spray test, in the case of M340, because it is a steel with a high concentration of carbon, 0.54%. In the case of M333, with carbon at 0.28%, the nitriding parameters can be adjusted to achieve a modified layer with high corrosion resistance, but at the cost of not achieving a very high resistance to wear compared to the material standard, where the relative mass loss was 0.75 while in the case of more layer it was 0.68. Still, the 0.75 relative loss would indicate that the wear resistance increased by 50%.

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2. Materials and Methods

The steel samples used were those with the AISI 431 code, the nitriding process was evaluated using 12 cylindrical specimens of an inch in diameter and 1 inch in height. The average chemical composition is shown in table 2.1. Nitriding times were cumulative from 5 hours to 15 hours. The specimens were nitrided in a pulsed DC plasma in a national heat treatment plant; The electric discharge is initiated and sustained in a low pressure gas mixture (10 mbar) with a voltage applied between the central, negative electrode, where the parts to be nitrided are located, and an external electrode that is a stainless steel cylinder that surrounds to the cathode; Ion nitriding was carried out in this experimental device in a nitrogen and hydrogen plasma (25% N2), for 5 hours and at a temperature of 360 °C. Prior to nitriding, cleaning was carried out by "sputtering" in an argon and hydrogen plasma to eliminate the passivating oxide and activate the surface. The preparation of the samples for hardness, micro-hardness and microscopy tests, included the steps of countering the new cutting faces, which were devastated with water-based sandpaper of N° 80, 120, 240, 320, 400, 600, 800, 1000 and 2000; finally, the polishing is carried out with 1µm and 0.3 µm diamond paste. To identify the nitrided layer in the AISI 431 stainless steel, an electrolytic polishing will be carried out in nital at 3%, in this way we will sensitize the steel and observe its microstructure and layer; For the microscopic evaluation, a DM600M series LEICA microscope was used. The hardness profile was obtained on the Vickers scale with a load of 1 gram.

Table 1. Chemical composition of AISI 431 steel

Chemical composition (%)						
C	Mn	P	S	Si	Cr	Ni
0.2	1	0.04	0.03	1	15-17	1.25-2.5

Table 2. AISI 431 steel mechanical properties

Mechanical properties			
Tensile strength (Kg/mm^2)	Elastic limit (Kg/mm^2)Min.	Elongation (mm)	Hardness (HB)
84 - 92	**65**	**15**	**238 - 280**

Fig. 1 (a) Cutting of AISI 431 steel samples, (b) plasma nitriding, (c) electrolytic etching of samples

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3. Results

3.1 Hardness profiles.

Figures 2 and 3, presents the average hardness data obtained, both the surface hardnesses, as well as those obtained through the cross section of the ionically nitrided AISI 431 steels at the temperature of 400 °C, at the times of 05, 10 and 15 hours. In Fig. 2, both the surface hardness values are presented, as well as their average data, while fig. 3, the values are shown both on the surface, as well as those achieved in the non-nitrided areas (base metal).

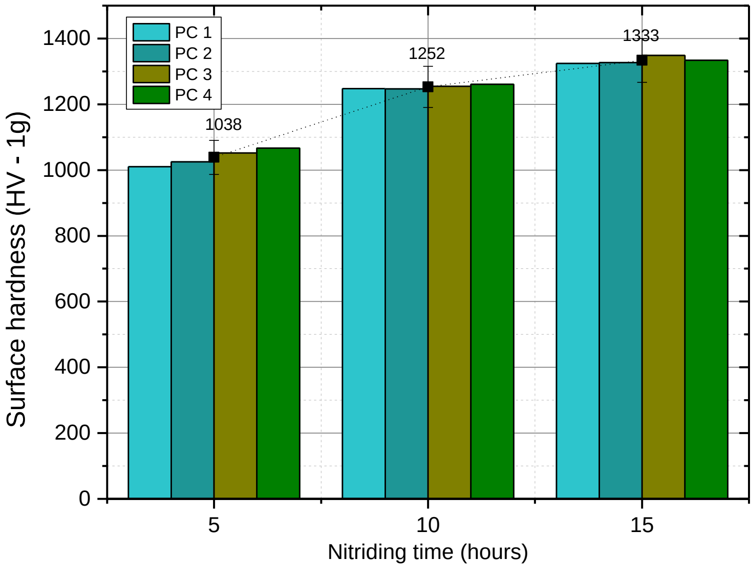

Fig. 2 Surface hardness obtained in each control, average surface hardness for ionically nitrided AISI 431 steel at 400 °C at 05, 10 and 15 hours of treatment

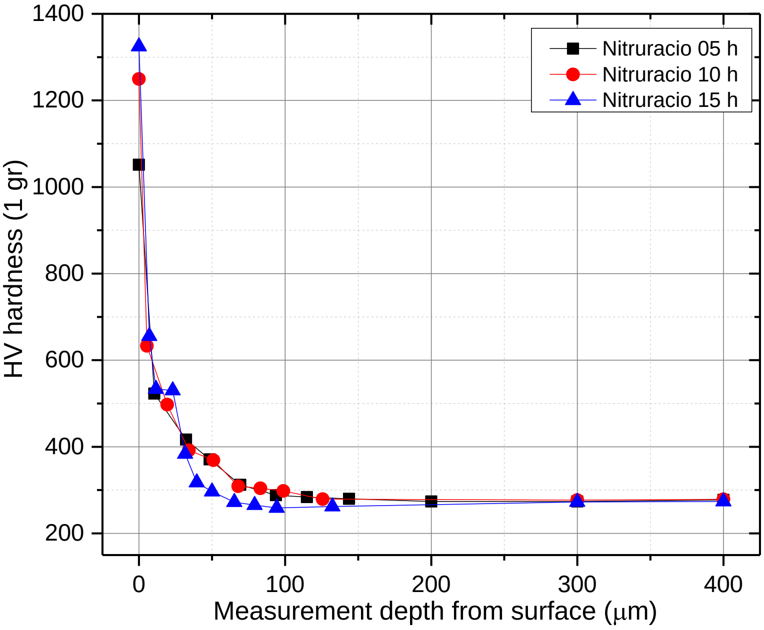

Fig. 3. Average hardness profile for ionically nitrided AISI 431 steel at 400 °C at 05, 10 and 15 hours of treatment

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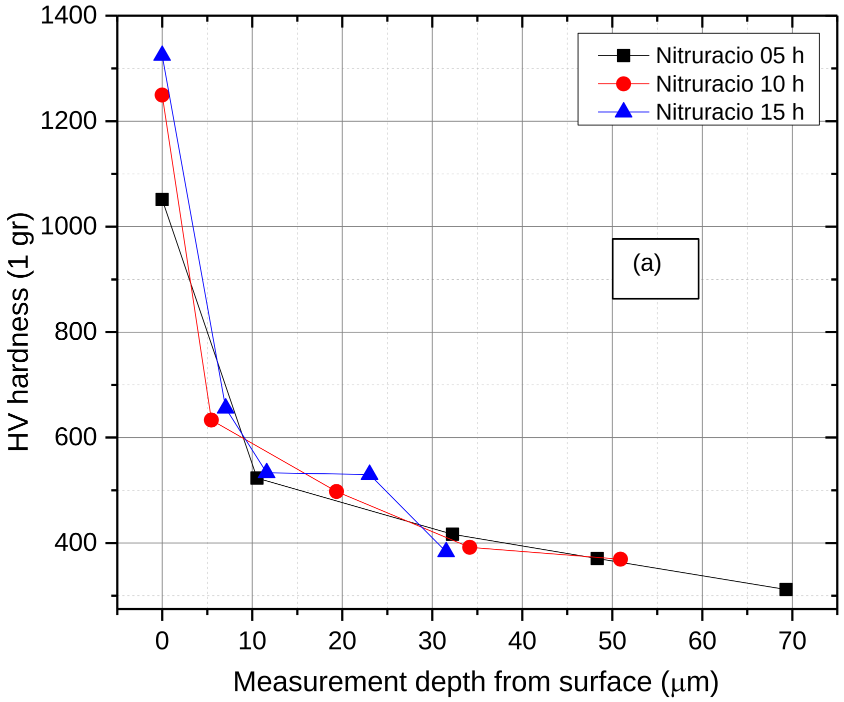
Figures 2 and 3 show the values of surface hardness and hardness profile obtained, from them it can be seen that after 15 hours of nitriding time for AISI 431 steel, at the ionic nitriding conditions used, the hardness values are achieved. higher being in this case 1333 HV (1gr), then there are the values of 10 hours and finally those of 05 hours of ionic nitriding, the hardness values reached being 1252 and 1038 HV respectively. Likewise, the hardness value of the core, in this it almost remained almost unchanged, despite the difference in residence times in the oven, although we must remember the low temperature of the process (400 °C), which is probably the cause of this constancy of hardness data in the core. In addition to the aforementioned, the smallest difference between the maximum of the hardness values of 10 and 15 hours and the maximum difference between the hardness data reported between 05 and 10 hours is evidenced, the cause of which is probably the decrease in capacity diffusion of nitrogen, due to the nitride film previously formed and of greater thickness as a function of nitriding time, since diffusion through said layer is more difficult than diffusion in the absence of it. Fernandes and others (2010) reached similar conclusions, who in the conclusions of their research work mention that the treatment parameters such as temperature and time are the determinants of the characteristics of the nitrided zone, the surface hardness depends on these variables. , depth of hardened area, layer thicknesses, giving a lower formation kinetics, as the treatment time increases due to the variation in diffusion constants, due to the type of nitride that could be formed both on the surface and in the subsurface zone of the nitrided zone. Fig. 4 (a, b) shows the trend in the measurement of the depth of the hardened zone, assuming the plant parameter of 400 HV (Value taken from process control) as the lower limit of the hardened zone in the case of the nitriding process This is how it can be seen what is described in the tables and figures in the appendices, on the trend towards uniformity of the ionic nitriding process.



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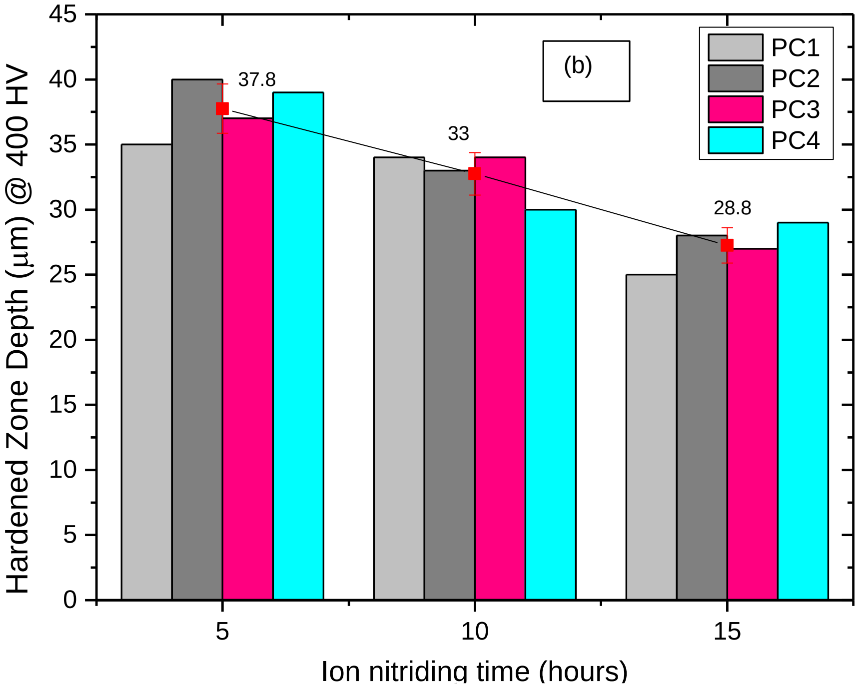
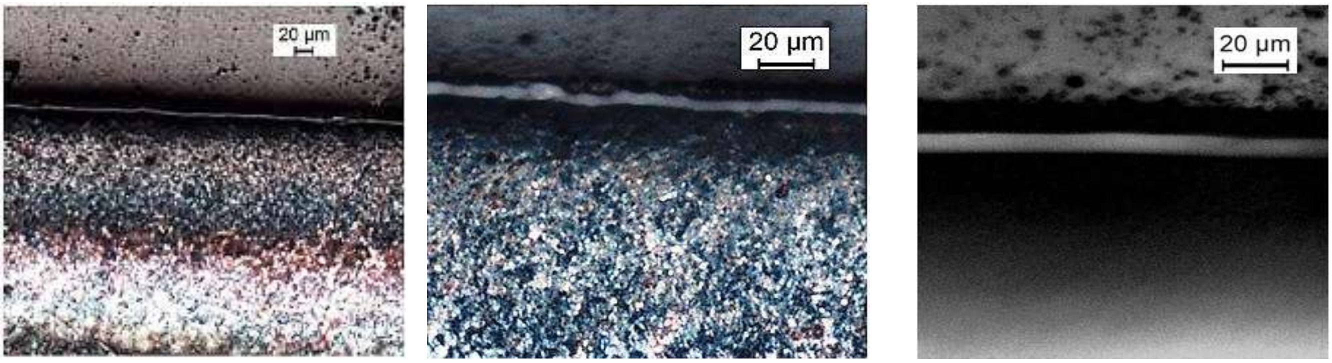


Fig. 4 (a) Average hardness profile for ionically nitrided AISI 431 steel at 400 °C at 05, 10 and 15 hours of treatment, indicating the depth zone considered effectively hardened. (b) Data on the depth of the effective hardened zone, indicating average values at each level of study evaluated.

3.2 Metallographic Evaluation

Fig. 5 shows what was obtained in the metallographic inspection of the surfaces of the AISI 431 steel samples nitrided in ionic form at different treatment times, at the proposed study levels.



(a) (b) (c)

Fig. 5. Photomicrographs obtained in the metallographic evaluation in the surface areas of ionically nitrided AISI 431 steel at 400 °C at 05 (a), 10 (b) and 15 (c) hours of treatment. 500 X.

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From fig. 6, we can appreciate the effect of nitriding time, versus the thickness of the nitride layer formed, thus we see the directly proportional effect of time with respect to the mentioned thickness, the average values of layer thicknesses are shown in fig. 6. If we make a comparison of the trends found in fig. 4 and 5, we can see the trend of inversely proportional between them, that is, the thickness of the white layer increases with the decrease in the depth of the hardened effective layer (400HV).

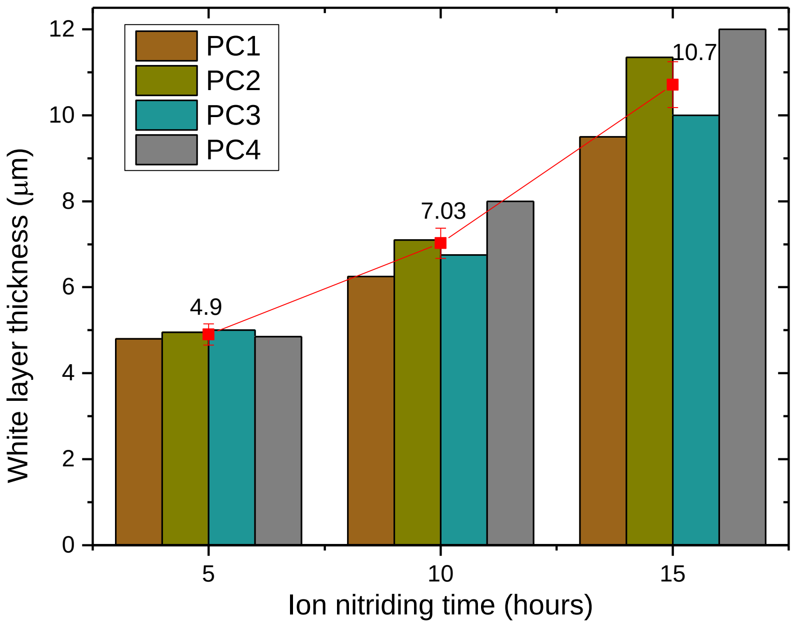

Fig. 6. Nitride layer thicknesses formed during the ionic nitriding process of AISI 431 steel at the proposed study times

If we now graph the data obtained as a summary we can show the following figures. Fig. 7 shows the trend of nitriding time values, obtained surface hardnesses and effective layer depth, fig. 8 shows the trend of graphing the nitriding time data, average maximum hardnesses obtained and nitride layer thicknesses. Finally fig. 9, Plots the trend of nitriding time, effective layer thickness and white layer thickness. From figures 4, 6, 7, 8 and 9 we can say that the difference of greatest implication when varying the ionic nitriding temperature for plasma nitrided AISI 431 steel in the times of 05, 10 and 15 hours, is the surface hardness and white layer thickness, since the core hardness data, hardened zone depth (400 HV), are almost constant, this would be due to the low treatment temperature used (400 °C), temperature in the which the process of diffusion and formation of nitrides is relatively low compared to nitriding processes in gases (almost 600 °C), which only makes noticeable differences in surface hardness and this in turn would be given by the thickness of nitride layer formed, as shown in fig. 6 similar conclusions reached by Miche and others (1995), in it they also mention the advantages and effects of the low temperatures of the ionic nitriding process, among them, thin layer thicknesses, low variability of the core hardness (non-hardened zone), the most affected parameters being the thickness of the white layer and the surface hardness.

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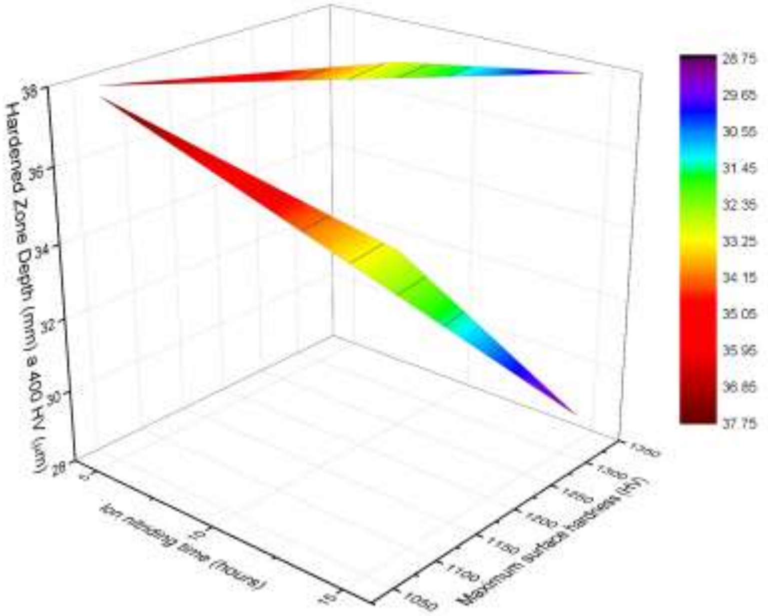

Fig. 7. Data trends for nitriding time, maximum surface hardness, and effective layer depth, for AISI 431 steel ionically nitrided at 400 °C at 05, 10 and 15 hours of treatment.

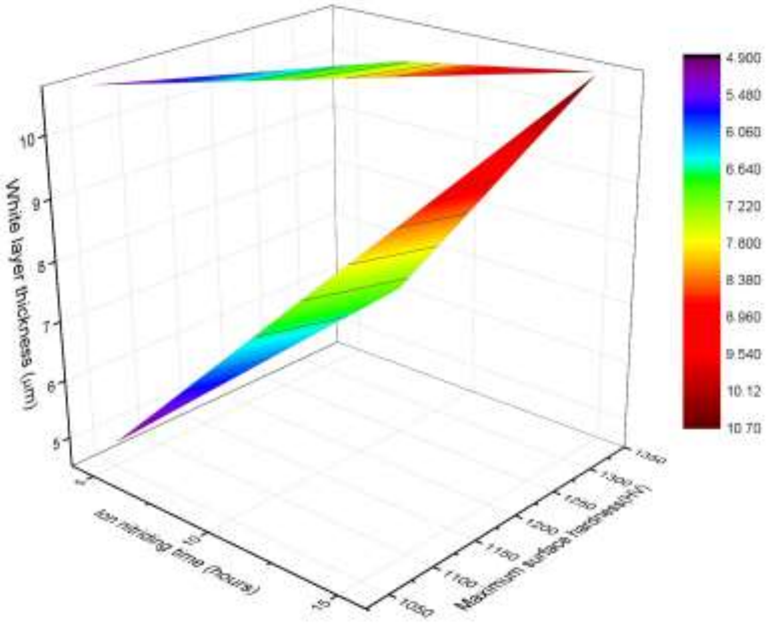

Fig. 8. Data trends for nitriding time, maximum surface hardness, and white layer thickness, for AISI 431 steel ionically nitrided at 400 °C at 05, 10 and 15 hours of treatment

Figures 7, 8 and 9 clearly show the inverse relationship of the white layer thickness and the effective layer thickness, in addition to the direct relationship of the treatment time and the nitride layer thickness and the inverse relationship of the nitriding time and the effective layer thickness. In addition to the aforementioned, there is the fact that the plasma nitriding process is the effective way of nitriding stainless steels, such as AISI 431 steel, and that the hardness of the nitride layer is dependent on its chemical composition, time of nitriding and the procedure used, thus in this case and as mentioned by Matthews et al. (1995), chromium is the second main nitride former and therefore is responsible for the hardness obtained on its surface, in addition to The same work indicates the effect of the formation of the nitride layer, indicating that the formation of this layer decreases by a factor of between 100 to 1000 the nitrogen diffusion coefficient in the matrix phase due to the effect of said layer; thus the same effect is expected in the growth of the nitride layer with increasing time, thus, in this way, what is reported in fig. 3.1 regarding the difference in maximum hardness in the intervals from 10 to 15 and from 05 to 10 hours of treatment.

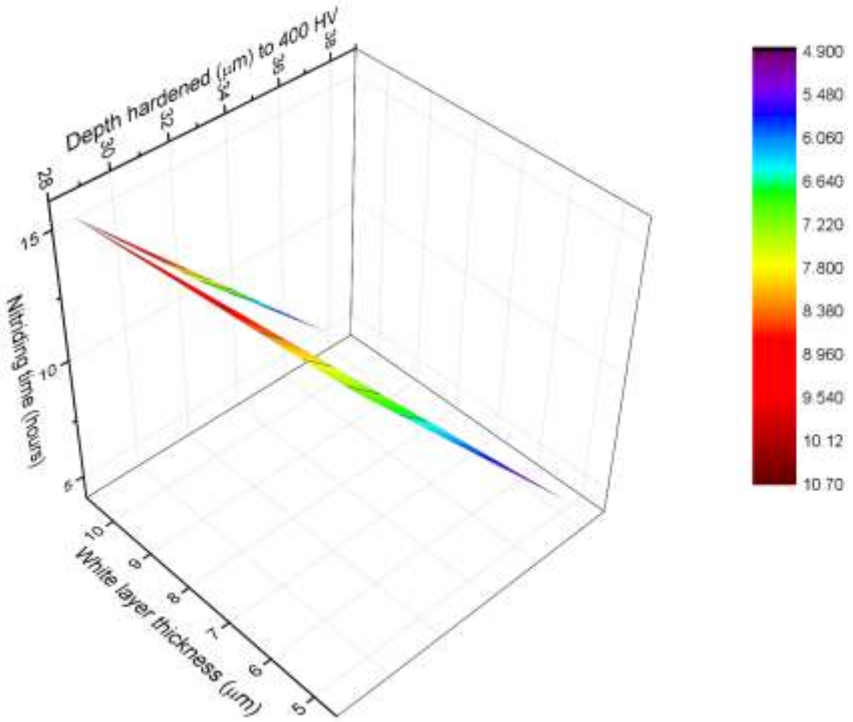

Fig. 9. Trends in the data of nitriding time, effective layer depth and white layer thickness, for AISI 431 steel ionically nitrided at 400 °C at 05, 10 and 15 hours of treatment.

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3. Conclusions

- Increasing the plasma nitriding time in the range of (5 - 15) hours, increases the surface hardness values from (1038 to 1333) HV-1g, respectively, for the case of AISI 431 stainless steel.
- The increase in plasma nitriding time in the range of (5 - 15) hours, achieves low differences in the thickness of the hardened layer at 400 HV-1g, in all evaluated cases it was on average 30 µm.
- Increasing the plasma nitriding time in the range of (5 - 15) hours, increased the nitride layer thickness (white layer) from 4.09 µm to 10.7 µm, respectively, for the case of AISI 431 stainless steel.

Acknowledgments

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