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TECCIENCIA

Characterizing the Mechanical Properties of Steel AISI-SAE 4140 to Apply a Plasma Nitriding Process

Caracterización de las Propiedades Mecánicas de un Acero AISI-SAE 4140 al Aplicarle un Proceso de Nitruración por Plasma

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Abstract

Surface treatments give physical and chemical properties to the materials, without them showing any change in their dimensions. Plasma nitriding is a thermochemical treatment, which increases some mechanical properties of metallic materials. In this article the characterization of the mechanical and micro-structural properties of an AISI-SAE 4140 steel is presented. To carry out the metallographic characterization, stress, hardness, abrasive wear and X-ray diffraction tests, the AISI-SAE 4140 steel was mechanized and prepared delivered in a hardened state (quenched and tempered) produced by Shah Alloys Ltd. making test pieces with their corresponding ASTM standards and nitrifying by plasma half of these at 500ºC for 10 hours. A thickness of 6.78 μm was obtained from the nitride layer that the material acquired. A microstructural constitution was found formed by martensite and austenite retained in the base material, maintaining the same structure with the thermochemical process. The nitrided surface showed in its atomic chemical composition (At%) the presence of 8.04% N, 1.69% O and 1.38% Al (element of great importance to achieve great hardness), revealing an increase of 18.55% of surface hardness and 78.87% of wear resistance. Regarding the tensile properties, there is a 77.65% decrease in its elongation when subjected to medium and low tensile loads, presenting a deformation of 0.09 mm for the last stress effort of 600 MPa.

Keywords: Characterization, Steel, Nitriding, Plasma, Tension, Hardness, Wear, Optical metallography, Thermochemical treatment.

Resumen

Los tratamientos superficiales aportan propiedades físicas y químicas a los materiales, sin que estos presenten cambio alguno en sus dimensiones. La nitruración por plasma es un tratamiento termoquímico, con el cual se aumentan algunas propiedades mecánicas de los materiales metálicos. En este artículo se presenta la caracterización de las propiedades mecánicas y micro-estructurales de un acero AISI-SAE 4140 Para la realización de la caracterización metalográfica, los ensayos de tensión, dureza, desgaste abrasivo y difracción de rayos X, se mecanizo y preparo el acero AISI-SAE 4140 entregado en estado bonificado (templado y revenido) producido por Shah Alloys Ltd. Realizando probetas con sus correspondientes normas ASTM y nitrurando por plasma la mitad de estas a 500ºC durante 10 horas. Se obtuvo un espesor de 6.78 µm de la capa de nitruros que adquirió el material. Se encontró una constitución microestructural formada por martensita y austenita retenida en el material base, manteniéndose la misma estructura con el proceso termoquímico. La superficie nitrurada mostro en su composición química en átomos (At%) la presencia de un 8.04% N, 1.69% O y 1.38% Al (elemento de gran importancia para lograr grandes dureza), revelándose un incremento del 18.55% de dureza superficial y un 78.87 % de la resistencia al desgaste, con respecto a las propiedades a tensión hay una disminución de un 77,65% su elongación al ser sometido a cargas medianas y bajas de tracción, presentando una deformación de 0.09 mm para un esfuerzo último de tensión de 600 MPa.

***Palabras clave*:** Caracterización, Acero, Nitruración, Plasma, Tensión, Dureza, Desgaste, Metalografía óptica, Tratamiento termoquímico.

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1. Introduction

Technological advance and the demand for materials used in different types of industries at the national level, has made steel strong in the country as a raw material for the production of machine components. The mechanical requirements imposed on steel sometimes are of greater proportion than those it can provide; therefore, the use of various surface treatments or coatings becomes necessary, thus contributing to the dimensional stability and surface hardness [1].

Plasma nitriding is a method applied to metallic materials, which seeks to increase mechanical properties modifying the physical and chemical properties in the surface layer, without any change to their dimensions. Basically, it consists in the application of a nitrogen environment, subjected to a voltage differential in order to ionize the gas forming the plasma and iron nitrides that eventually will be disseminated on the surface of the exposed material [2].

The AISI/SAE 4140 steel is widely used in industries such as those of polymers, casting elements, metallurgical, extrusion processes, wire drawing, among others. Usually, a material is subjected to high mechanical stress and therefore must possess high dimensional stability, toughness, abrasive wear resistance and suitable hardness. The nitriding process has been applied to this steel previously and it has been analyzed only through surface hardness and metallography, but lacking all the tension and wear resistance studies, not reflecting all the mechanical properties that can be provided to the material with the application of plasma nitriding.

The main purpose of this research is the study of the interaction of the thermochemical nitriding treatment upon the mechanical properties of hardness, wear resistance and an AISI/SAE 4140 steel ductility by applying mechanical laboratory tests such as Vickers hardness, wear resistance and tensile stress under the guidelines of the standards ASTM E92, G65 and E8, respectively; contrasting the original condition with the nitrided condition, to validate the application of this process, and allowing to complement the mechanical study of the material, thus expanding the knowledge on the phenomenon of plasma nitriding.

2. Materials and methods

The rationale of the development of the research was conducted by compiling books, theses, journal articles, guidelines, laboratory, mechanical testing, and web pages for the search of the different physical, mechanical and chemical concepts that are related. The experimental methodology is presented below.

(a)

(b)

Figure 1 Test specimens. (a) Tension. (b) Abrasive wear.

Figure 2 Test specimen prepared for metallographic test.

Figure 3 Olympus GX-71 metallographic microscope.

Figure 4 Plasma nitriding equipment NitrEos of Tratar S.A. Available at: http://www.tratar.com.co

2.1 Planning of mechanical testing and preparation of test specimens

Plasma nitriding interferes in a small surface layer of the material. For that, tests were established and test specimens were made for abrasive wear, Rockwell hardness, and tension, considering the ASTM Standards ASTM G-65 (Standard Test Method for Measuring Abrasion Using the Dry Sand/Rubber Wheel Apparatus), ASTM E18-03 (Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials) and ASTM E8M-04 (Standard test method for tensile tests of metallic materials), respectively

For the tensile test, eight cylindrical test specimens were made, as it can be seen in Figure 1. For the abrasive wear test, eight rectangular test specimens were made with the following dimensions (width of 1 in, length of 3.5 in and thickness of 1/8 - ½ in), taking into account that the face that will confront the material with the rubber wheel and dry sand should be smooth and flat with minimum porosity and cracking as shown in Figure 1.

2.2 Materials of thermal insulation

For the microstructural characterization the guidelines of the ASTM E3-01 (Standard Practice for Preparation of Metallographic Specimen) were followed. Mirror-polished samples were prepared, using a polish table having a polish cloth soaked in abrasive substances.

Polishing was performed three times, first using alumina or aluminum oxide as the abrasive to 3 µm to 0.3 µm and finally to 6 µm diamond paste. One of the samples can be seen in figure 2. The chemical attack was used with nitric acid or Nital at 2% with an attack time of 3 seconds.

The metallographic analysis was performed by means of the use of the inverted microscope Olympus GX 71, in order to identify steel metallographic phases that are subject of study in original or delivery condition. The application of the plasma nitriding process was made in the TRATAR S.A. facilities, located in the city of Medellín. The machine NitrEos, shown in figure 4, was used. For this process, the parameters of duration of 10 hours and 500°C of temperature were applied.

2.3 Optical microscopy and X-ray diffraction

Optical microscopy was performed in order to analyze the depth of the nitrided layer of the material. This was observed in the metallographic microscope of the Technological Units of Santander.

Figure 5 Hardness tester THBRV – 187_5D.

Figure 6 Universal testing machine. Microcomputer Controlled WDW-20E.

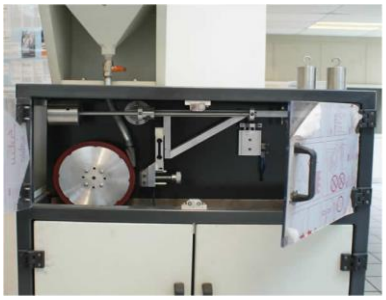


Figure 7 Abrasive wear machine.

Figure 8 Observation of the nitriding process through the pyrex viewer.

In addition to optical microscopy, the method of Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) was carried out with the microscope of the Universidad Industrial de Santander branch Guatiguara.

2.4 Mechanical tests

Characterization of the mechanical properties of the AISI/SAE 4140 steel when applying the plasma nitriding process and making the respective analysis was established with the tests of Rockwell hardness, wear resistance and tension.
The tests were performed under two conditions: A (test specimens without the plasma nitriding process) and B (test specimens with plasma nitriding process).

The Rockwell hardness and stress mechanical tests will be made on the equipment respectively shown in figures 5 and 6 of the Material Resistance Laboratory of the Technological Units of Santander and the wear resistance test is developed in the Pontifical Bolivarian University, through the equipment that can be seen in Figure 7, whose principle of operation is based on a small motor that spins a chlorobutyl rubber wheel at a certain rpm, which is covered by a curtain of dry sand through a nozzle that connects to the hopper of sand, and a static support system in which the test specimen is placed and produces the contact of it with the wheel. This causes the loss of mass of the test specimen when exposed to contact the wheel and the flowing sand.

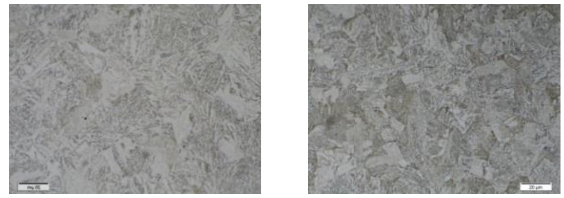
3. Result and Analysis

3.1 Plasma Nitriding

The plasma nitriding process was performed in a 10-hour period with a temperature of 500°C. Figure 2 shows the image of the material through the pyrex viewer of the NitrEos reactor when the process was applied, where the abrasive wear test specimens can be seen covered by a violet color plasma.

3.2 Plasma Nitriding

The structural formation of the material as a steel with martensite and remaining austenite is remarked. Figures 9 (a) and (b) show the AISI/SAE 4140 steel micrographs, nitrided and non-nitrided, where it is added that the phases of the material remained unaltered after the application of the thermochemical process, because the temperature employed in the process was 500ºC, at this temperature the nitrogen diffusion occurs only in the ferrite phase of the material, preventing any metallographic phase change and presents a null deformation of the piece treated.



(a) Non-nitrided specimen (b) nitrided specimen

Figure 9 Metallography of test specimens A and B with 2 % Nital treatment, magnification 500X.

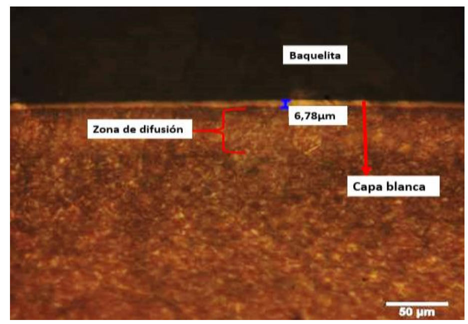


Figure 10 Thickness of the nitrided layer.

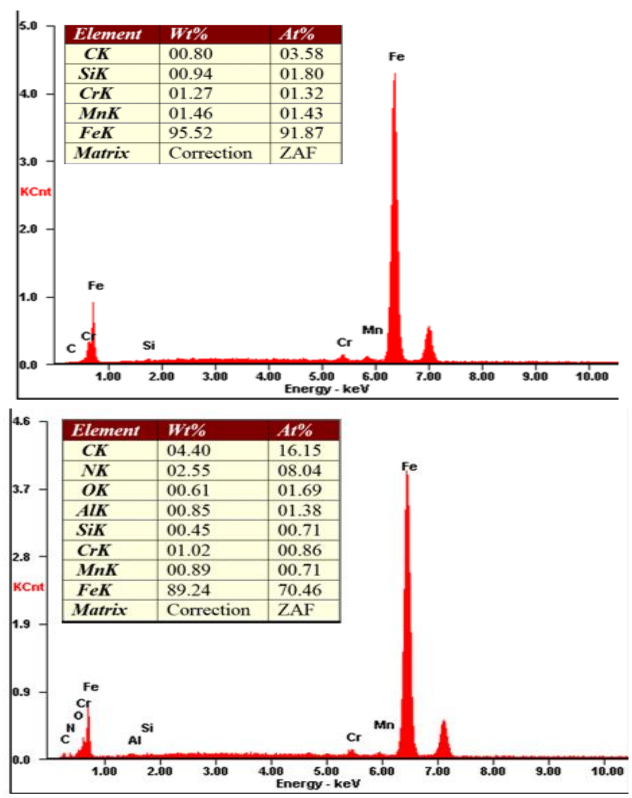


Figure 11 SEM analysis of EDX spectrum. (Above: Non-nitrided steel. Below: nitrided steel).

3.4 Analysis of the mechanical properties of the AISI-SAE 4140 steel.

3.4.1 Hardness test.

Through the implantation of nitrogen atoms in the surface layer of the material, produced by diffusion during the plasma nitriding process, an 18.55% increase was obtained in the hardness of the nitrided material with respect to the non-nitrided one, as observed in table 1 and figure 13. This increase in hardness is due to the conglomeration of elements such as iron, nitrogen, carbon and small amounts of oxygen.

3.4.2 Abrasive wear test.

The mechanical behavior of the surface of the nitrided material subjected to friction, was analyzed by means of the abrasive wear test, to the nitrided and non-nitrided steel AISI SAE 4140, in three times, measured in number of revolutions (200, 1000 and 6000), where an imminent increase in the wear resistance of the material was observed of 78.87% at 200 rpm and at 6000 rpm a contribution of 40,5% in the wear resistance for the nitrided material. This was due to the decrease of the friction coefficient, produced by the fine and dispersed precipitates of iron nitrides as observed in the micrography of the nitrided test specimen (see table 2). Additionally, tables 3 and 4 present the results obtained for the nitrided and non-nitrided samples, respectively.

Table 1 Results of the Rockwell hardness test.

	Rockwell hardness C Test (RHC)	
	Non-nitrided steel	Nitrided steel
	50.2	58.4
	48.7	58.6
	49.4	58.8
average	49.43	58.6

Table 2 Test grooves size from the abrasive wear test (non-nitrided test specimens – nitrided test specimens – 200 revs – 2000 revs – 6000 revs).

	Probetas sin nitrurar	Probetas nitruradas
200 revoluciones		
1000 revoluciones		
6000 revoluciones		

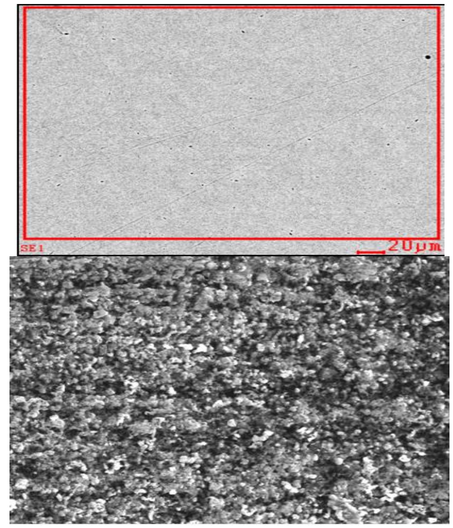


Figure 12 Micrograph 10000X.

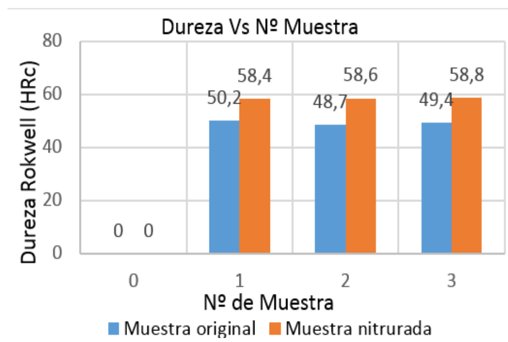


Figure 13 Contrast hardness test for the original sample and nitrided sample (Rockwell hardness (HRc) – sample No. – Hardness vs. sample No.- original sample- nitrided sample).

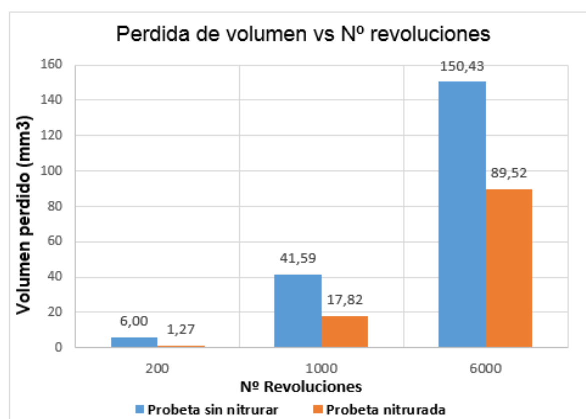


Figure 14 Volume loss vs. number of revolutions. (Volume loss (mm3) – no. of revolutions – non-nitrided test specimen – nitrided test-specimen).

Table 3 Results of the abrasive wear test, non-nitrided test specimens.

Dry sand/rubber Wheel test ASTM G5 – Process 1			
MATERIAL REFERENCE	AISI 4140 STEEL	AMOUNT:	3
VOLUME LOSS AVERAGE:	66.0048667 mm^3	VARIATION COEFFICIENT	0,5671%
TEST DATA			
Material description	AISI 4140 STEEL	Wheel diameter	9 in
Thermal treatment	hardened	Wheel width	0.5 in
Rockwell hardness	49.43 HRC	Wheel speed	200 rpm
Test number	A	B	C
Test load (lbf):	30	30	30
Wheel revolutions:	6000	1000	200
Sand flow(g/min):	320	320	320
Initial mass (g):	180.7739	177.7425	177.4194
Final mass (g):	179.6053	177.4194	177.3728
Lost mass (g):	1.1686	0.3231	0.0466
Density (g/cm3):	7.7686	7.7686	7.7686
Volume loss (mm3) (lost mass/density *1000):	150.4261	41.5905	5.998

Table 4 Results of the abrasive wear test, nitrided test specimens.

Dry sand/rubber Wheel test ASTM G5 – Process 1			
MATERIAL REFERENCE	AISI 4140 STEEL	AMOUNT:	3
VOLUME LOSS AVERAGE:	36.2013667 mm^3	VARIATION COEFFICIENT	0,5671%
TEST DATA			
Material description	AISI 4140 STEEL	Wheel diameter	9 in
Thermal treatment	Hardened and plasma nitrided	Wheel width	0.5 in
Rockwell hardness	58.6 HRC	Wheel speed	200 rpm
Test number	A	B	C
Test load (lbf):	30	30	30
Wheel revolutions:	6000	1000	200
Sand flow(g/min):	320	320	320
Initial mass (g):	179.4386	180.9556	180.8178
Final mass (g):	178.7463	180.8178	180.8080
Lost mass (g):	0.6923	0.1378	0.0098
Density (g/cm3):	7.7336	7.7336	7.7336
Volume loss (mm3) (lost mass/density *1000):	89.5185	17.8184	1.2672

The tensile stress test was carried out with a 20 KN load and at 2 mm/min speed until rupture. A notorious change in the properties of the AISI-SAE steel was evidenced, where the decrease of the yield stress due to the presence of the small elastic zone of the nitrided material and a notorious decrease in the ultimate tensile strength and the stress rupture were observed, the last two being of the same magnitude as indicated by the maximum normal stress theory (see Figures 15 and 16).

(a) Non-nitrided test specimen, ductile fracture (b) Nitrided test specimen, brittle fracture

Figure 15 Fracture of the test specimens in the tensile stress test.

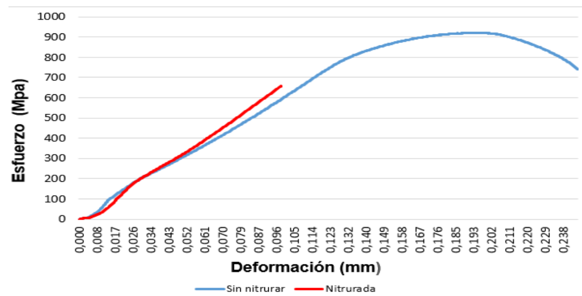
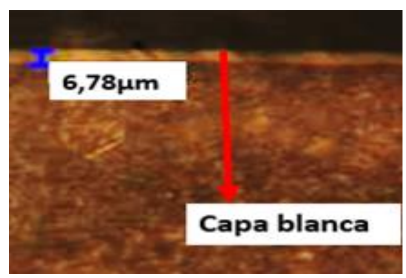


Figure 16 Stress v. strain curve (Stress (Mpa) – strain (mm) non nitrided- nitride).



(a) Plasma nitriding, 10 h 500 ° C

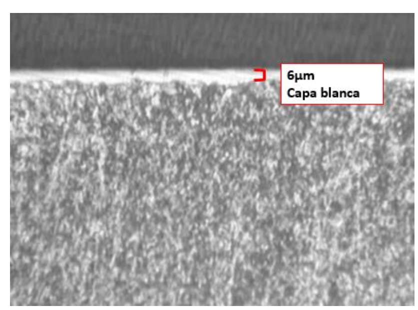


Figure 17 Validation of the nitrided layer against other authors.

3.5 Validating results against bibliographic sources

The results of the plasma nitriding process to the AISI-SAE 4140 steel for 10 h at 500ºC were compared with previous studies applying the same thermochemical treatment to the same material, varying some of the operation conditions. One of these variations can be seen in Figure 17.

4. Conclusions

The present research revealed that the AISI-SAE 4140 steel in a delivery status is constituted by a structure of martensite and remaining austenite, of low impurity and with complex observation due to its high austenization temperature in the quenching and tempering to alleviate the material's thermic tensions.

The 6.87 µm iron nitrides layer formed the plasma nitriding process is constituted by the interaction of 2.55% nitrogen and 0.61% oxygen diffused in the surface of the steel, revealing in turn the presence of 0.85% aluminum that was not present in the chemical composition of the non-nitrided material due to its low content, which showcases the increase in hardness of the nitrided layer.

The plasma nitriding increased the wear resistance of the material under study by 78.87%, due to the 18.55% increase in the hardness of the material's surface layer constituted by iron nitrides.

The plasma nitrided (500ºC 10h) AISI-SAE 4140 steel decreased its elongation under medium and low tensile loads by 77.65%, presenting a 0.009 mm deformation for an ultimate tensile strength of 600 Mpa, where no fracture propagation is still produced in the material's surface layer.

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