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CHEMICAL CARACTERIZATION OF 4140 STEEL IMPLANTED BY NITROGEN IONS[1]

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Abstract
AISI-SAE 4140 sample surfaces of different roughness which are implanted by nitrogen ions of 20 keV and 30 keV at a dose of 10^{17} ions/cm^2 through a three-dimensional ion implantation technique are studied. Crystal phases of nitrogen compositions of the implanted samples, obtained with help of an x-ray diffraction method, are confronted with the data reported by the *International Centre for Diffraction Data* (ICDD), PDF-2. It is observed that the implanted into the metal nitrogen atoms produce changes in orientation of crystal planes that is manifested as variations of the intensity of the refracted rays and of cell dimensions (a displacement of 2 theta of the maximum intensity position). An analysis for determining nitrogen atoms implanted by high-voltage pulsed discharges at low pressures in the crystal structure of the solid surface was carried out by X-Ray Diffraction due to this technique permits to assess the possibility of formation of new compounds.
Keywords: Ion Implantation; Superficial treatment; Corrosion; Roughness.

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1 INTRODUCTION

The three-dimensional ion implantation process for high-voltage pulsed discharges at low pressures is to change (no change in the geometric dimensions) the properties occurring in physical and chemical surface and subsurface layers of materials. With these amendments seek to improve some of its characteristics, becoming a subject of great interest in the study of materials science.

Reported in 1997 by Vladimir Khvesyuk and Petr Tsygankov[1] a new method of implementation via ionic plasma which is not required prior preparation of a basic plasma. With this method the implantation process is performed in a pulsed high voltage discharge at low pressures,[2,3] lit in the left branch of Paschen curve. This method is called ion implantation 3DII dimensional self-sustained discharge is obtained which is characterized by a small thickness of the cathode fall region and the great stability of the plasma during the download process. One consequence of these features is the monoenergetic ion spectrum, a normal angle of incidence of ions and a significant increase in ionic flux density, compared to other methods using ion beams. This is very important since the maximum dose retained by introducing a sample is determined by the angle of incidence of the ions and the sputtering coefficient of the surface.[4] Additionally, the range of low pressure significantly decreases the possibility of contamination of samples.

Current research points to a growing number of applications that can benefit from ion implantation. There is no doubt that the progress of this branch of industry is primarily linked to successes in the development of corresponding devices. Extensive studies in the processes of implementation allowed tribological properties produce materials with predetermined according to a specific need. In the production of such materials requires advanced equipment, such as dispositive JUPITER (Joint Universal Plasma and Ion Technologies of Experimental Reactor).[5-7] The JUPITER has been built and designed under the direction of Dr. Valery Soap Dougan, especially for the investigation of high-voltage pulsed discharges at low pressures and for the treatment of metal surfaces.

The completion of this research is developed in four stages. The first step is the choice of material, design, manufacture and preparation of samples. The second step is to perform chemical characterization of base material in order to verify the study material (AISI SAE 4140). The third stage development surface treatment by ion implantation of nitrogen in the surface area of the samples. The fourth step is to perform chemical characterization by means of a qualitative analysis of the present phases or compounds by comparison with the profile observed diffraction profiles reported in the database PDF-2 of the International Centre for Diffraction Data (ICDD). Finally, raised the conclusions of this research.

2 SAMPLE PREPARATION

According to a preliminary study that seeks to provide solutions to industrial problems where the material basis of many elements are produced in steel AISI SAE 4140[8,9] in the present investigation was designed and produced disk-shaped samples whose dimensions are shown in Figure 1.

With the purpose of studying the behavior of the surface, the specimens were carried different slab surface with abrasive paper of SiC number 320, 600, 1200 and 6μ and 1μ cloth according to ASTM E 3-95, in order to have samples with different roughness. The goal is to eliminate the rough surface layer dis-twisted metal and

take into account the effects of contamination of samples generated by the adsorption of atoms and molecules and the formation of mono-layers in the environment.

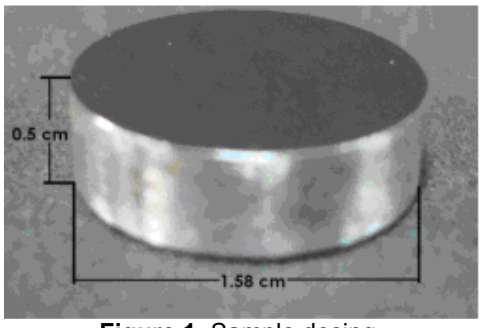


Figure 1. Sample desing.

3 CHEMICAL COMPOSITION OF THE SAMPLES

The chemical composition analysis of samples by optical emission spectroscopy (EEO), confirm the type of base material used in the manufacture of the samples. The results (see Table 1) indicate that the base material of the samples correspond to an alloy steel with chemical composition similar to AISI SAE 4140, according to the values reported by ASTM A 322.

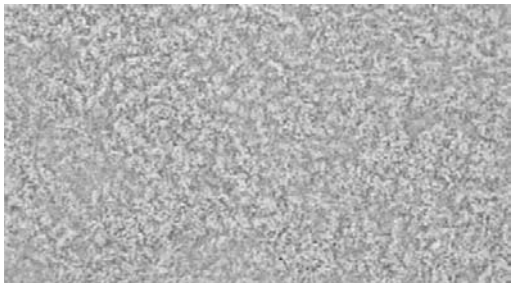
Table 1. Chemical composition of the simple tested AISI 4140 steel

ELEMENT	%	ELEMENT	%
C	0.40	Cu	0.17
Cr	0.85	Sn	0.019
Mo	0.14	V	0.026
Mn	0.85	Al	0.002
Si	0.24	Ni	0.09
S	0.018	Nb	0.007
P	0.012	Cu	0.17

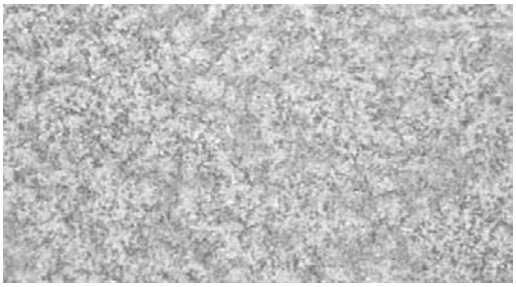
4 METALLOGRAPHIC ANALYSIS

With the metallographic analysis seeks more information from the structure of the material under test (SAE AISI 4140), for this purpose using an Olympus microscope OBM X3, where they will have information about the structure of the study material.

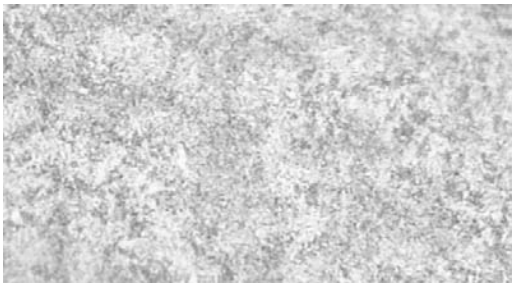
Metallographic analysis shows (Figure 2) a microstructure of tempered martensite, which proves to me that the samples are AISI SAE 4140 steel quenched state (quenched and tempered).



a) Microestructure at 100x zoom.



b) Microestructure at 200x zoom.



c) Microestructure at 500x zoom.

Figure 2. Microestructure of the samples tested. Zoom to: a) 100x, b) 200x and c) 500x.

5 SURFACE TREATMENT SAMPLES

Ion implantation of nitrogen that runs in this paper builds on previous research results which were able to establish the range of discharge voltages, the method of determining the dose of implanted ions and the gas pressures of work.

Within the discharge chamber (see Figure 3) samples are located where these function as cathode and the walls of the chamber functions as the anode, when starting the download, some samples generates a non-uniform plasma density almost zero and around the anode is formed a plasma known as anode.

Figura 3. Samples are cathode and anode walls.

As working gas nitrogen is used (N_2), that due to a potential difference gives rise to the discharge which generates the anode plasma which is responsible for providing nitrogen ion cathode fall zone, which produces almost the entire drop voltage applied to the discharge.

The process of ion implantation of nitrogen in the samples is performed in a pulsed unloading at low pressures with voltage of 30 kV, 0.40 ms pulse duration,

pulse repetition frequency of 30 Hz and a 60-minute exposure treatment an average surface pressure of 0.4 Pa.

The implanted dose on the surface of the samples was determined by taking into account the value of the coefficient of secondary ion-electron emission (γ) and total current value obtained during the surface treatment of the samples. The literature reports a secondary emission coefficient γ=1 in a discharge of 30 kV during a process of ion implantation in 304 stainless steel cathode. Data in Table 2, are recorded total current values for the samples with polished surface with a cloth of 1μ and 6μ.

Table 2. Total current for the treatment of the samples

Voltage discharge	Polished Surface	Total Current [A]
20kV	Sandpaper 320	0.28
20kV	Sandpaper 600	0.32
20kV	Sandpaper 1200	0.317
20kV	Cloth 6μ	0.359
20kV	Cloth 1μ	0.324
30kV	Cloth 6μ	0.678
30kV	Cloth 1μ	0.648

With the data of total current and secondary emission coefficient values are obtained ion current, ion current density and ion flow. The dose of nitrogen ions implanted on the surface of the samples were obtained through the mathematical expression (1), it replaced the value of the calculated ion flux and parameters of treatment:[10,11]

$$D = 2\Phi f dt \qquad (1)$$

where D is the dose of implanted ions, Φ the ion flux incident to the sample, f the pulse repetition frequency, d the pulse duration, t the time of implantation and number two is that the molecules of nitrogen gas are diatomic.

Data in Table 3, is show the ion concentration of nitrogen with a cloth surface finish and implanted at energies of 30 KeV.[12]

Table 3. Nitrogen doses implanted at energies of 30 keV

Polished Surface	Nitrogen doses [ions / cm^2]
Sandpaper 320	$3.4632 * 10^{17}$
Sandpaper 600	$3.9576 * 10^{17}$
Sandpaper 1200	$3.9220 * 10^{17}$
cloth 6μ	$4.4409 * 10^{17}$
cloth 1μ	$4.0089 * 10^{17}$

Table 2 shows that the samples treated at energies of 30keV in particular the surface finish to cloth 6μ a higher discharge current. As for those that are treated to the highest energies of 20keV discharge current is present on samples with a cloth surface finish and less common for those with a sandpaper surface finish 320.

As shown in Table 3, samples with higher nitrogen doses are given to those found with polished surface 6μ

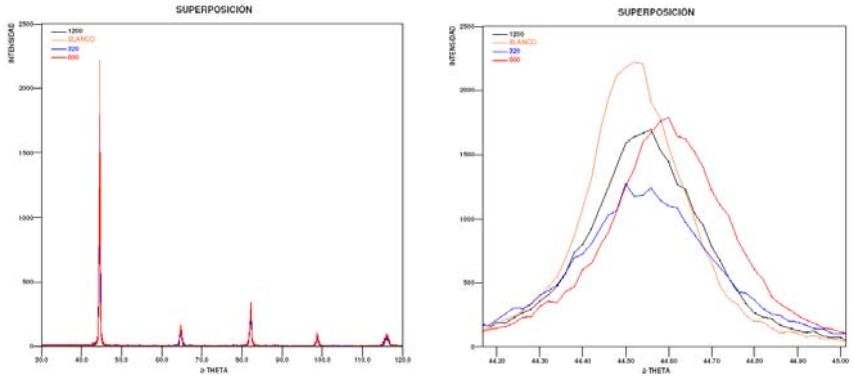
5 CHEMICAL CHARACTERIZATION

Chemical characterization was performed using the X-Ray diffraction technique (XRD) in the X-Ray diffraction laboratory of the Universidad Industrial de Santander. Samples without the deployment and implemented which have different surface finish were performed a qualitative analysis of the phases present or compound by comparing the observed profile with the profiles reported in the diffraction database PDF-2 of the International Centre for Diffraction Data (ICDD) (see Figure 4 and 5).

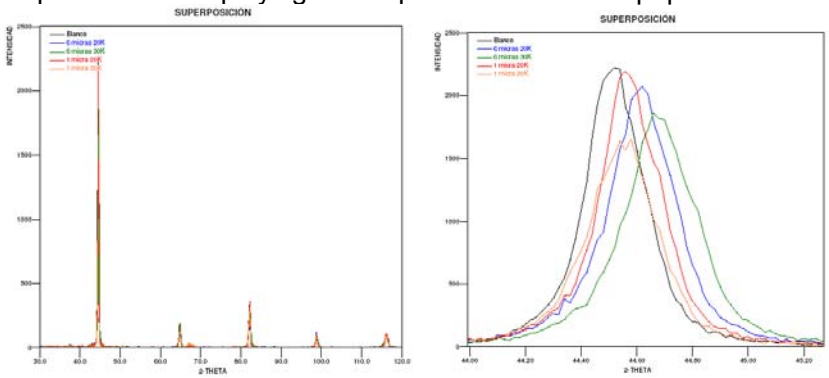
The samples were mounted on an aluminum sample holder using the frontal filling technique and the analysis was performed in a Rigaku powder diffractometer model D/MAX IIIB under the conditions reported in Table 4.

Table 4. Working conditions of the XRD

Parameter	Value
Voltage	40kV
Current	20mA
Slit DS	1º
Slit RS	0.3mm
Slit SS	1º
Sampling	0.02º 2-Theta
Measuring Range	30º-120º 2.Theta
Radiation	$CuK_{\alpha 1}$
Monochromator	Graphite
Type of measure	Continuos
Scanning speed	1.2º/min



a) Samples without deploying and implanted with a sandpaper surface finish



b) Samples without deploying and implanted with a cloth surface finish

Figure 4. Overlap the spectra obteined by XRD.

From the analysis obtained by XRD technique and represented in Figure 4 (a and b) is not observed the formation of a compound such as iron nitride (Fe_xN), which may be due to its location in a small percentage and the low sensitivity technique does not substantiate or nitrogen to be implanted in the metal is located in the interstitial spaces is shielded by another element or compound.

However, when performing the overlapping of the samples, it is clear that the nitrogen in the metal causes changes such as decreased levels refracted in the range of intensities and the reduction of the unit cell in the position of 2-Theta.

6 CONCLUSIONS

An analysis of chemical composition and metallographic to the samples before being subjected to surface treatment, where it is found that the base material under study correspond to an alloy steel SAE 4140 AISI state subsidized micro-structure of tempered martensite.

Device is achieved in three-dimensional surface modification JUPITER uniform samples of AISI SAE 4140 with parameters of frequency, pulse duration and voltage set during the experiments. The voltage and current pulses have a quasi-rectangular shape over the total dicharge power.

The X-Ray diffraction technique reported interesting results regarding the effect captive propelled by ion implantation of nitrogen in the sample SAE AISI 4140 steel which results in decreased levels refracted in the range of intensities and reducing surface unit cell at position 2 theta compared with the sample without the deployment.

Acknowledgements

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