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Effect of surface roughness on nitriding processes by dielectric barrier discharges of carbon steel

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Abstract. Changes in the behaviour of steels to carbon, for three different finishes, surfaces were determined to be nitride by atmospheric pressure dielectric barrier discharge. The surfaces were characterized both morphologically and structurally before and after the treatment. The techniques used were: measurement of roughness and electrochemical testing (Tafel). Results show changes differential depending on the type of finish, but in any case, an improvement of corrosion resistance.

1. Introduction

Most of the industrial companies are affected by the increase of the stops of machinery and the costs of maintenance due to the change of the pieces with wear. It is claimed that surface treatments are one of the best techniques to increase the life cycles of a mechanical part [1] and to improve some properties such as corrosion resistance, fatigue resistance, even to increase hardness [2].

Nitriding is one of the most advanced surface treatment methods of research and development in recent years. Ionic nitriding is a process of thermochemical diffusion with which the formation of a hard surface layer is obtained by absorption and diffusion of nitrogen [3]. To improve the conditions of the surface layer, it is said that it depends on the application and use of the dielectric barrier discharge method for the formation of a uniform nitride layer [4]. This treatment allows to improve the properties of the steel so that it presents a better performance in different mechanical parts, such as axles, pinions, cutting tools or other industrial application [5].

Studies on nitriding processes and electrochemical tests are generally performed without regard to surface finishes [3].

The finishes of the pieces in the industry do not have surfaces with a type of mirror polished, then the techniques developed in prototypes cannot be implemented at a productive level, this is known from the perspective of fractal geometry, an object whose structure is irregular can have a behaviour in which the effective surface is greater than the geometric, thus varying the parameters considered in the estimations of surface densities [6], so that for this investigation the effects of the roughness of the material were determined, the nitriding was performed three types of surface finishes and electrochemical tests, such as polarization curves (Tafel) and corrosion rate.

2. Materials and methods

2.1. Preparation of samples

Samples were taken $2.54 cm$ in diameter and $1 cm$ in height. The cleaning of surface oxides and impurities of the specimens and the roughness profile were achieved by means of an abrasive blast

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called sand blasting. This process was done in a private laboratory of blasting services. The anchor profile (roughness level) was given according to the European standard SIS 05 5900 for degree of rust A and finish SA 3 or SSPC SP5 according to the American standard. The roughness measurement was done by means of a digital surface roughness tester SRT 6200. The test pieces were cleaned with isopropyl alcohol by means of ultrasound to discard residues of other materials, before nitriding.

2.2. Nitriding of test pieces

The nitriding was done by means of a reactor composed of the electrodes (aluminium anode and as cathode each steel sample) with a tempered glass dielectric barrier of $3.0 mm$ thickness under a nitrogen atmosphere of 99.9% purity. The assembly was made using Teflon and Mica accessories insulators. The dielectric barrier was maintained in contact with the anode at a distance of about $1.0 mm$ from the cathode in a nitrogen atmosphere. Figure 1 shows the nitriding device used in this research.
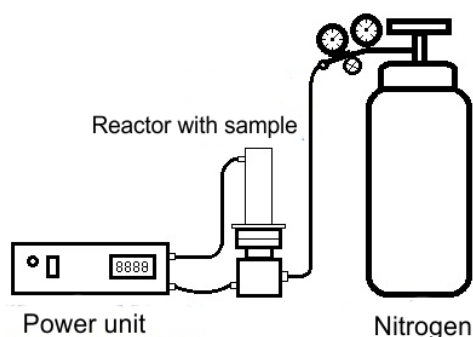

Figure 1. Dielectric barrier nitriding equipment.

The nitriding process of the samples had an average duration of 32 minutes. First, the nitrogen passage is opened to displace the reactor air for five minutes. Subsequently the nitriding discharge is done for 15 minutes with a gas pressure in the pipes of $12.5\,psi$. Then the unit is turned off and the gas flow is left open for five minutes. Finally, it takes seven minutes for the nitrogen tank gauges to reach zero. The flow measured by displacement with grade five high purity nitrogen is $3.6\,liters/hour$. The quality of nitriding depends on the flow rate of hydrogen, which has an optimum value [7].

2.3. Electrochemical tests

One of the most widely used electrochemical methods in scientific and practical characterization studies is the polarization curves method [8]. These tests are done to measure the corrosion rate of the samples, before and after the nitriding. An electrochemical cell was used, the GAMRY software version 6.30 of the Echem Analyst TM package was used for data acquisition and processing. The devices or electrochemical cells used in this type of test basically consist of five elements: container, probe holder, working electrode (test tube connected to potentiostat), reference electrode (standard to measure electrochemical effects) and auxiliary electrode. Figure 2 shows the assembly of the device which was started by placing the steel sample in the propeller holder which is secured to the acrylic structure (A). The sample was tightened with screws to press on a rubber (B). 3% sodium chloride solution is injected into the cell container, avoiding the presence of bubbles (C). After reaching the top of the solution, the reference electrode (silver electrode) with direct connection to the data processor (D) is inserted into one of the holes in the vessel. The resulting connectors are matched to the colour and the assembly is placed inside the protective plastic housing.

For the measurement of the corrosion rate, the Echem Analyst TM software reports the results on Tafel with its respective data Table. The polarization curves allow to find values of the anodic and cathodic slopes. The parameters found in the polarization curves enable corrosion current densities and corrosion rates to be found in milliseconds per year (mpy) [9].
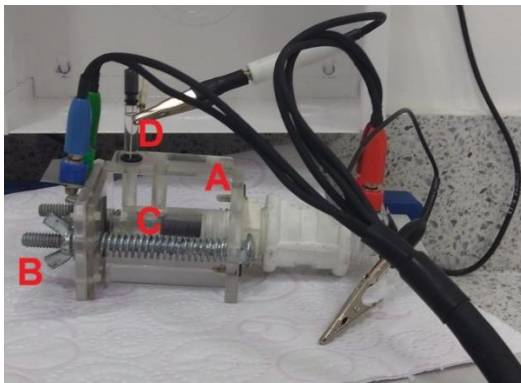
Figure 2. Assembly of the specimen. (A) Acrylic structure, (B) screws that press the sample. (c) Cell container and (D) reference electrode (silver electrode) with direct connection to the data processor.

3. Results

One of the important and relevant results is the change in the value of the roughness after the nitriding process by means of a dielectric barrier discharge, and it is presented by the roughness test 2 as shown in Table 1 with a value of $6.15\mu m$. The dielectric barrier discharges are characterized by the formation of micro arcs, which are directly linked to the type of topography of the surface of the electrodes and the surface of the dielectric material that separates them [6]. As a consequence of the above, the roughness specimen 2 presents changes in its surface structure as the higher peaks are devastated and allow a significant reduction in the value of its roughness.

Table 1. Comparison of roughness before and after nitriding.

Measurements	Without nitriding	With nitriding
Roughness machining	$0.58\mu m$	$1.32\mu m$
Roughness 1	$3.72\mu m$	$3.87\mu m$
Roughness 2	$6.38\mu m$	$6.15\mu m$

Table 2 shows the results of the corrosion rate in each of the test specimens, where the samples with roughness 1 present the best behaviour with a value of $3.238 mpy$. And Figure 3 shows the comparative graph of each of the Tafel curves.

The results show that the method of nitriding by means of discharge of dielectric barrier at atmospheric pressure is an effective method to improve the mechanical properties and to obtain greater resistance against the phenomenon of corrosion [10], and experimental results indicate that the rapid nitriding process not only improves the nitriding rate remarkably but also maintains the high hardness of the nitride layer [11].

Table 2. Corrosion analysis of samples of Tafel curves.

| Samples	Machined without treatment	Machined with treatment	R1 without treatment	R1 with treatment	R2 without treatment	R2 with treatment
E_{corr}(mV)	-547.0	-545.0	-612.0	-581.0	-570.0	-533.0
I_{corr}(μA)	20.40	18.20	14.50	7.09	20.20	13.70
Beta A (V/decade)	$82.80e^{-3}$	$238.0e^{-3}$	$101.2e^{-3}$	$78.4e^{-3}$	$84.8e^{-3}$	$109.1e^{-3}$
Beta C (V/decade)	$619.9e^{-3}$	$82.00e^{-3}$	$585.8e^{-3}$	$60.70e^{-3}$	$551.8e^{-3}$	$180.4e^{-3}$
Corrosion rate (mpy)	9.345	8.319	6.608	3.238	9.226	6.244
Chic square	341.0e-3	207.1e-3	467.7e-3	564.6e-3	286.0e-3	183.8e-3
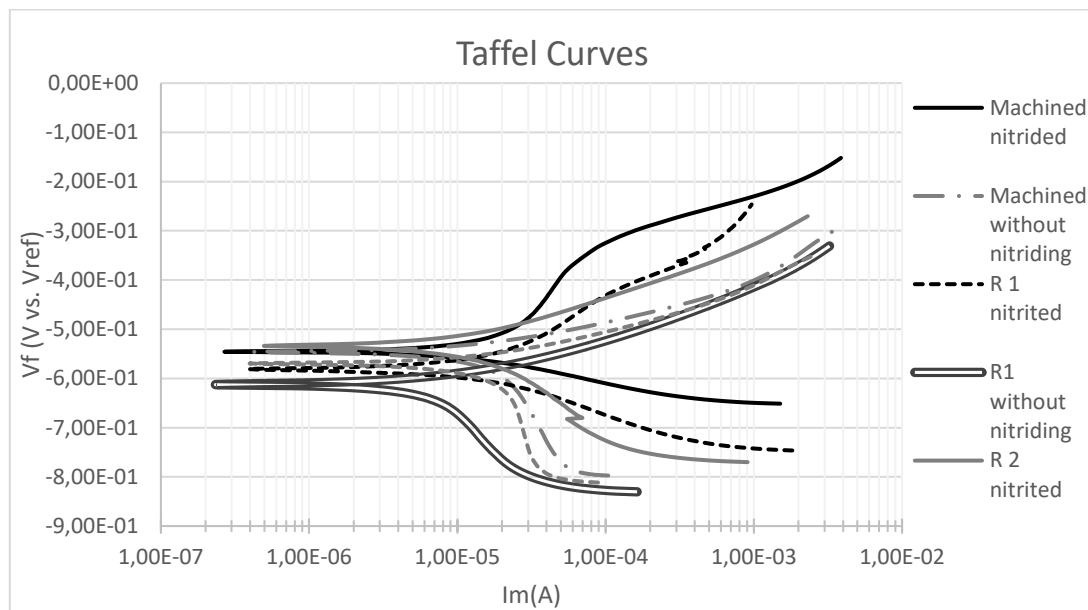
Figure 3. Comparative graph of Tafel curves in each of the samples.

4. Conclusions

From the investigation, it is concluded that the corrosion rate of the AISI/SAE 1020 steel is lower when a surface nitriding process has been done, but the corrosion rate is further reduced in the samples with a greater roughness, machined sample decreases to a very small amount because its surface layer does not exist some chemical elements that allow a greater reduction in its speed of corrosion. It should be noted that the test specimens of the blasting process reduce the corrosion rate without performing a nitriding process due to the content of some elements and the action they exert in the microstructure of the AISI/SAE 1020 steel, which will be used for future studies.

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