



Corrosion resistance of bainitic steels alloyed with boron

Resistencia a la corrosión de los aceros bainíticos aleados con boro

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ABSTRACT

Keywords:

steels with boron,
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Due to its good combination of properties and its wide range of application in the automotive and rail sector, among others, bainitic steels have been studied by modifying their chemical composition using different alloys in order to improve their resistance, as is the case of boron, which is an element that allows to increase the mechanical properties of steel and its behavior against phenomena such as wear when is used in small quantities. In the present investigation, through of saline chamber and potentiodynamic polarization tests, the influence of boron was studied in steels with bainitic microstructure against phenomena as corrosion by varying its percentage in each sample analyzed, which was developed from the measurement of parameters such as the percentage of corroded area and the current density. Although no significant influence of boron was observed in the saline chamber results, in potentiodynamic polarization, a greater reduction in current density was observed for steel with a higher boron content, which results in a better resistance to corrosion compared to other steels that had a lower percentage of this element.

RESUMEN

Palabras clave:

aceros con boro,
bainita,
corrosión.

Debido a su buena combinación de propiedades y a su aplicación en el sector automotriz y ferroviario, entre otros, los aceros bainíticos han sido objeto de estudio al modificar su composición química mediante el uso de diferentes aleantes con el fin de mejorar su resistencia, como es el caso del boro, el cual es un elemento que permite incrementar las propiedades mecánicas del acero y su comportamiento frente a fenómenos como el desgaste al ser utilizado en pequeñas cantidades. En el presente trabajo, por medio de ensayos de cámara salina y de polarización potenciodinámica, se estudió la influencia del boro en aceros con microestructura bainítica frente a fenómenos como la corrosión al variar su porcentaje en cada muestra analizada, lo cual se desarrolló a partir de la medición de parámetros como el porcentaje de área corroída y la densidad de corriente. Aunque en los resultados de cámara salina no se observó una influencia significativa del boro, en los de polarización potenciodinámica se apreció una mayor reducción en la densidad de corriente para el acero que contenía mayor porcentaje de boro en su composición, lo cual indica una mayor resistencia a la corrosión frente a los aceros que tenían un menor porcentaje de este elemento.

Introduction

Research on steels with bainitic microstructure has been carried out since the 1920s (20th century), where a new microstructure was discovered by Davenport and Bain, which differed from perlite and martensite, which was

named bainite in honour of Bain in 1934 [1]. Since the discovery of bainite, steels with a variety of designs in their chemical composition have been manufactured and marketed at an industrial level, such as in the automobile and railway sectors, among others [2].

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Bainitic steels present within their microstructure a decomposition of austenite where a pre-bainitic phase is reached in the transformation stage [3]. Kinetics starts with the nucleation at the austenitic grain boundary of a bainite subunit, from which new subunits of this microconstituent are continuously nucleated. Unlike the displacive phase transformation, the evolution of bainite is accompanied by a process of carbon diffusion from the ferrite plates to the surrounding austenite when performed at high temperatures, originating upper bainite, while if this diffusion is generated at low temperatures, the precipitation of carbides within the ferrite occurs, originating the lower bainite [4].

Steels with bainitic microstructure have been manufactured from processes such as hot-rolling and alloying elements such as cobalt and aluminum have been used, in quantities of less than 2%, to accelerate the transformation of their microstructure [5]. In addition, silicon has been used between 1.5 and 2% to control cementite precipitation during the bainitic transformation process [6].

The addition of boron has a pronounced effect on the microstructure, where the segregation of this element at the grain boundaries decreases the nucleation of proeutectoid ferrite and the iron diffusion coefficient. The effect of boron on a bainitic steel will be transcendental when the precipitates of this element do not form at the grain boundaries of austenite, improving the hardenability of the material [7].

In bainitic steels a variety of studies have been developed in order to analyze their resistance to corrosion. Kazum, Kannan, Beladi, Timokhina and Khoddam [8] compared the resistance to this phenomenon of a nanostructured bainitic steel and a martensitic one, using techniques such as potentiodynamic polarization. The results indicated a decrease in current density of 85% of bainitic steel against martensitic, because bainitic steel had a selective austenite solution retained by the high carbon distribution in this phase during its transformation process, taking into account that the added silicon prevents the formation of carbides in the ferrite-bainitic, while martensitic steel indicated localized and intragranular corrosion.

In the research developed by Qu, Pang, Wang and Gao [9], the corrosion behavior of a two-phase low carbon steel, which was composed of ferrite and bainite, was analyzed.

Among other aspects related to the electrochemical behavior, Rosalbino, Scavino and Mortarino [10] evaluated the corrosion process in bainitic mold steels, finding a controlled diffusion reaction in the cathodic region while in the anodic region an active dissolution occurred, due to an increase in current density.

Wang, Li, Guan, Chen and Pu [11] performed a comparison of the corrosion resistance of a bainitic steel with an outdoor steel in a test where the specimens were exposed to a saline environment (sodium chloride). Corrosion resistance was calculated from the weight loss in each steel, measured after the test, which showed similar resistance to this phenomenon in both steels. In the microstructural analysis it was considered that a homogeneous microstructure of the bainitic steel effectively reduced the corrosion process.

In this work, bainitic steels with a boron content of 0, 20, 40 and 60 parts per million were manufactured, in order to analyze the influence that this element has on corrosion by means of the use of techniques such as potentiodynamic polarization and salt chamber. Subsequently, the characterization of the corroded surface was carried out by scanning electron microscopy (SEM) to study its corrosion products.

Materials and Methods

Steel Production

The manufacture of the steels was carried out in an induction furnace containing an alumina crucible (Al₂O₃) with a capacity of 8 kg, using a maximum power of 75 kilowatts, a variable frequency of 0 to 9000 Hertz and a temperature of approximately 1600 degrees centigrade. In this crucible, first the 1020 and 1045 steels were added and then the ferroalloys referring to ferrosilicon, ferrochrome, ferromanganese, ferromolybdenum, ferrovanadium and ferroboration, in the respective order. The molten metal was poured into a mold made of two A-36 steel L-profiles. The total weight for each alloy was approximately 7.5 kilograms.

For the purpose of identifying the steels to be tested, according to their boron content, the samples were identified as 0B, 20B, 40B and 60B. Table 1 presents a summary of the weight percentage value of parts per million of boron in each alloy.

Table I. Percentage of Boron within each alloy

Parts per million boron (PPM)	Weight percentage (%)
0	0
20	0,011
40	0,021
60	0,032

Thermomechanical treatment

The steel blocks, obtained from the casting process, were placed in a muffle furnace at a maximum operating temperature of 1200°C for one hour to homogenize their microstructure. Subsequently, the blocks were hot-rolled in a rolling mill with a reversal of rotation, in order to obtain a homogeneous grain structure. The temperature in the rolling process was monitored from a maximum value of 1100°C to a minimum of 900°C, to ensure that the deformation process took place in the austenite zone. The blocks were then cooled in air to room temperature and the specimens were obtained by a cutting process.

Microstructural and chemical characterization

The microstructural characterization was performed in a FEI QUANTA 200 scanning electron microscope. The micrographs were developed in secondary and retro-dispersed electron mode, with a high vacuum pressure of 3×10^{-7} torr and a voltage of 30 kV. Retro scattered electron images were achieved with a JEOL microscope, model JSM 6490-LV, in high vacuum and with a voltage of 10 kV. The samples were extracted from the laminated blocks and then their surface was polished and attacked with the 2% Nital reagent.

The structural characterization was performed by means of X-ray diffraction technique with a Bragg-Brentano X-Pert Pro Panalytical equipment. The measurements were developed with a monochromatic copper line $K\alpha$, wavelength 1.54 Å, current of 40 mA and potential difference 45 kV. The scanning range was 5° to 100° with step size 0.02° in continuous mode. The data was analysed by means of the X'Pert High Score Plus software.

Potentiodynamic polarization

Prior to the test, the surfaces of the specimens were polished with sandpaper up to 1500 grains per square inch and then cleaned for 5 minutes with isopropanol

and acetone in an ultrasonic immersion. The anti-corrosive properties of the steels were determined by the potentiodynamic polarization technique, taking as a reference what is described in the ASTM G3-89 [12] and ASTM G5-94 [13] standards. The test was carried out at room temperature in a Faraday cage using a Gillac potentiostat. The potential was controlled from -300 mV to 400mV with respect to that at rest, using a sweep speed of 30 mV/s. The curves obtained by the process were obtained with a scanning speed of 0.5 m/s.

During the test a three-electrode system was used, with bainitic steel as the working electrode. A calomel reference electrode and a high purity graphite counter electrode were also used. The steels were immersed in a 3.5% sodium chloride solution, which was prepared with distilled and deionised water. The area of the test tube exposed within the solution was 0.159 cm². Prior to the test the sample was stabilized within the solution for 30 minutes to allow for a stabilization of the resting potential.

Salt chamber

As described in the ASTM B117 [14] standard, tests were carried out in a salt chamber, in which a controlled corrosive environment was provided, to analyze the influence of boron on the corrosion resistance of bainitic steels. The surfaces of the samples to be tested were first polished with sandpaper up to 1500 grains per square inch and then degreased with 96% ethanol.

Inside the chamber, the steels were placed at an inclination of 15 to 30 degrees from the vertical and parallel to the direction of flow of the salt spray. An adequate distance between the test pieces was guaranteed in order to avoid contact between them, to allow the free circulation of the mist. Similarly, the installation of the test tubes within the chamber ensured an even distribution of the solution during the spraying cycle.

During the corrosion test, the temperature of the chamber was controlled between 34.8 and 35.2 °C, while the pH of the salt solution was kept between 6.5 and 7.2. The samples were exposed to the saline environment for a total period of 100 hours, however, the test was interrupted at 24, 48 and 72 hours to estimate the percentage of corroded area as a function of time. The experimental details used are summarized in Table 2:

Table II. Salt Chamber Test Variables

Reagents	Condensed salt solution	Cabin temperature
Distilled water with a conductivity of 4.21 uS/cm	Average volume of 27.75 ml.	Minimum 34.8°C
pH of 6,01	pH. minimum of 6.59 and maximum of 6.88	Maximum of: 35,2 °C.
Sodium chloride R.A.	Minimum specific gravity of 1.024 and maximum of 1.030.	

Results and Discussion

Microstructural characterization

Figure 1 shows the microstructure obtained from the steels, by means of scanning electron microscopy, after the thermomechanical treatment. In the micrographs it is seen that the microstructure was formed by small sheets of retained austenite that separate the subunits of ferrite-bainitic formed. The similarity in microstructure for each experimental unit is associated with the homogeneous manufacture of the blocks in the casting and hot-rolling processes, as described in the materials and methods section.

Figure 2 shows the diffractograms obtained by the X-ray diffraction technique for each steel. The presence of the BCC (centred body cubic) and FCC (centred face cubic) phases is considered. Likewise, from the peaks of each diffractogram a preponderant microstructure of ferrite (alpha iron) is observed, identified through the planes (110), (200), (211). The other phase present corresponded to retained austenite (gamma iron), associated with planes (111) and (200). The presence of martensite cannot be objectively stated in these results, because the diffraction peaks associated with this microconstituent are very close to the ferrite peaks, making it difficult to classify between the referred phases, which is in accordance with what has been analyzed by Bakshi and Santofimia [15], [16].

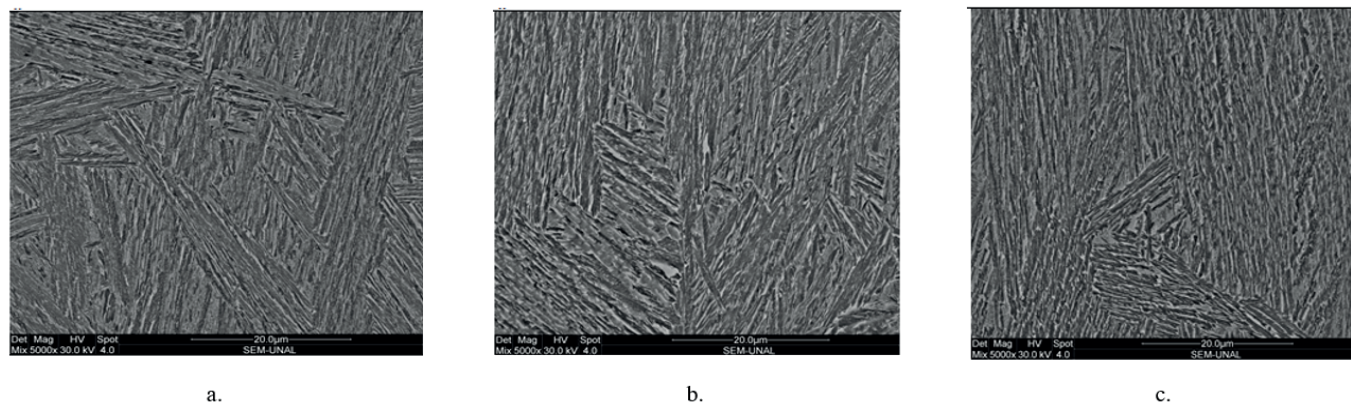


Figure 1. SEM micrographs of bainitic steels: a. 20B; b. 40B; c. 60B

In the diffractograms no peaks associated with carbides or cementite were observed, which indicates a possible reduction of these elements due to the presence of silicon, obtaining a bainitic microstructure free of carbides. On the other hand, within the diffractograms it was not possible to identify the presence of some precipitates referring to oxides, carbides and boron nitrides, which can be formed during the steel manufacturing process, which generally act as hardeners of the material [17]. However, for future work, a complementary study by transmission electron microscopy is recommended to identify possible carbides or borides, such as $Fe_23(C, B)_6$, which precipitate at the grain boundaries and which can facilitate the nucleation of proeutectoid ferrite, affecting both the hardenability and the toughness of the steel [18].

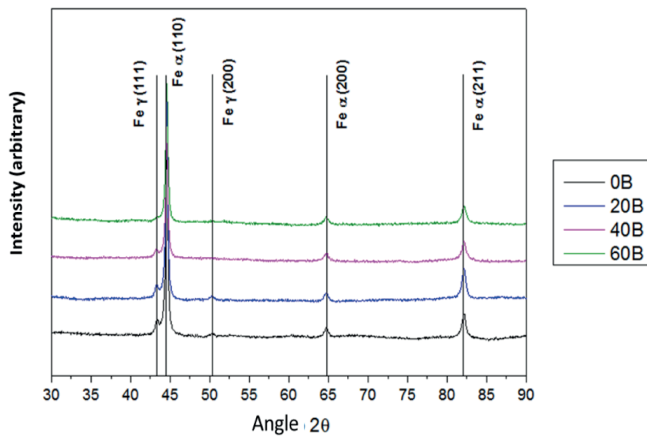


Figure 2. Diffractograms of bainitic steels with various boron contents

Figure 3 shows the approximate phase percentages for each steel. In general, there is a slight decrease in the austenite retained for steel 60B, which can be attributed to boron, since it favours the transformation rate and reduces the volume of unstable austenite and martensite, as shown by the research of Guerra, Bedolla, Mejía, Zuno and Maldonado [19], who considered this effect through metallographic analysis, associated with boron decreasing the transformation temperature of martensite. The research carried out by Bedolla, Guerra, Rainforth, Mejía and Maldonado [20] shows that boron is an element that increases carbon diffusion by accelerating the transformation, which generates a microstructure with a lower volume of unstable austenite and martensite.

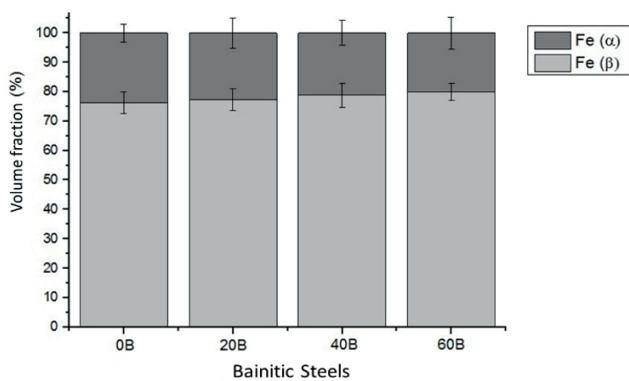


Figure 3. Approximate percentage of ferrite and austenite phases

Potentiodynamic “polarization Corrosion Test”

The mechanical properties and electrochemical corrosion

behaviour of steels depend on their microstructural arrangement [10]. In each phase there are microstructures and chemical compositions that can influence the development of a selective corrosion, which deteriorates the quality of the steel [9]. The steel detriment due to anodic dissolution is of vital importance in ferrous materials, since it originates in the grain boundary which is characterized by an amorphous region with high defect energy [21].

Tafel Curves

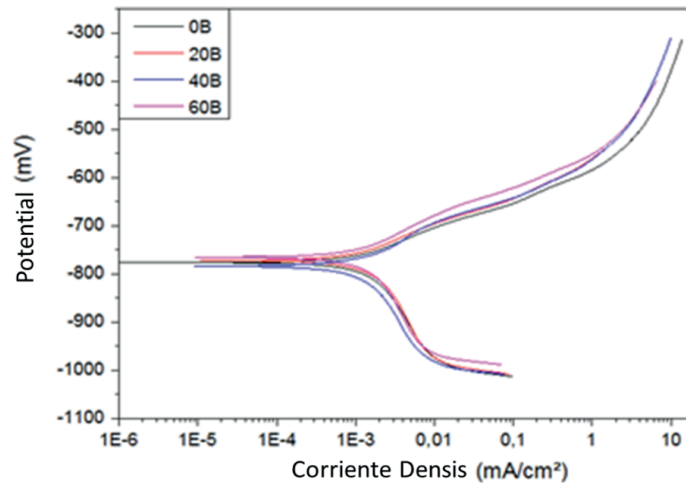
Figure 4 shows the results of the potentiodynamic polarisation tests for the steels produced, in a 3.5 % sodium chloride solution. The material with the lowest current density (indicating a lower flow of electrons from the steel surface to the electrolyte) and with a positive corrosion potential will have the highest corrosion resistance, promoting less material degradation. Steels with negative corrosion potential promote an abundant electrochemical action, which results in a greater predisposition to corrosion, accessing the release of more electrons to the electrolyte. These thermodynamic variables were calculated from Tafel's extrapolation technique.

Table 3 shows a summary of the electrochemical parameters evaluated in the curves obtained. It can be deduced that there was a decrease in the current density (I_{corr}) with the increase in the boron content, being the 60B steel the one that presented the lowest current density while in the 0B steel the highest value was obtained. This suggests that on the surface of steels containing more boron a more compact passive layer was produced, reducing the current flow with the electrolyte. The results do not show a significant difference in the corrosion potential of the steels, inferring that there is no correlation between the corrosion potential and the boron content.

Table III. Current density (I_{corr}) and corrosion potential (E_{corr})

Alloy	I_{corr} (mA/cm ²)	Standard deviation	E_{corr} (mV)	Standard deviation
0B	0,4342 ± 0,019	0,033	-756,04 ± 11,71	20,27
20B	0,3886 ± 0,003	0,004	-762,91 ± 4,6	7,96
40B	0,3812 ± 0,003	0,006	-779,93 ± 7,66	13,27
60B	0,3275 ± 0,033	0,057	-750,58 ± 25,1	43,46

On the other hand, in the steels tested, silicon was able to prevent the increase of carbides in the ferrite, preventing the dissolution process from originating in that phase due to the creation of small micro-galvanic cells, which is consistent with the study carried out by Moon, Sangal, Layek, Giribaskar and Mondal [22], who found the presence of these cells in the ferrite phase, associated with the presence of fine carbides, whose size is small in relation to the anodic region of the ferrite.

**Figure 4.** Potentiodynamic Polarization Curves for 0B; 20B; 40B; 60B steels

From the polarization curves (Figure 4) it can be seen that the cathode branch is related to the diffusion-controlled cathode reaction of the species in the electrolyte, e.g. the oxygen reduction reaction ($O_2 + 4H + 4e: 2H_2O$). Meanwhile, the anodic curves are dominated by a polarization activation, which characterizes an accelerated growth of current density, being applicable to its active dissolution. In some works, curves with approximately similar behavior were obtained [10], [23], who evaluated the corrosion resistance of bainitic steels for moulds and pipes, presenting in the curves a behavior associated to a controlled reaction of diffusion in the cathodic region and an accelerated increase of current density in the anodic region, which was associated to an active dissolution.

Analysis of corrosion products

Figure 5 shows the corroded surface of the bainitic microstructure in the sodium chloride solution. Scattered corrosion products on the surface of the steel and some cracks in the layers are observed, although the difference is not significant when taking into account the similarity in the microstructure for each steel. The layer that forms the corrosion products can provide an effective protection in the steels when it is compact, attributing the corrosion rate to the integrity of the mentioned products. The research developed by Du, Li, Liang, Liu, Jia and Cheng [24] shows that when the thickness of the layers increases over time, the corrosion rate of the steel can decrease. On the other hand, when the deposited layers are porous their structure will be weak, providing little protection to the steel.

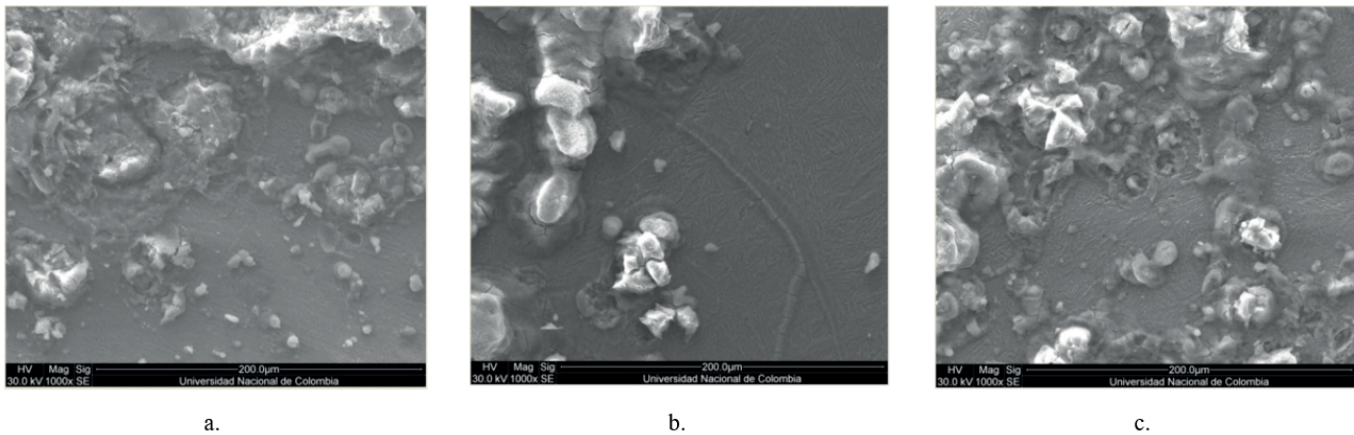


Figure 5. Corroded surface. a. 20B; b. 40B; c. 60B

Several factors could influence the development of the corroded surface, for example, one of them could be due to the ferrite which is characterized by the highest activity and from there the corrosion process could be carried out [9]. Similarly, a steel with a more refined bainitic microstructure contributes to a higher corrosion resistance [10], although this characteristic should not have been very noticeable among the steels tested, because the difference in corrosion resistance was not very significant. On the other hand, corrosion occurs in the surface defects of steels, such as at the grain boundaries or at the boundary of the bainitic ferrite phases, since they contain a high energy potential, dissolving anodically as a function of time in a solution with chlorine ions, whose steel surface is covered with corrosion products, such as metal oxides. Hakan et al. [21] have shown that in the grain boundaries a fast and aggressive reaction to chlorine ions forming pits occurs, being compatible with the shown in figure 5.

Figure 6 shows the elementary study of the chemical composition on the corroded surface by means of EDS spectra. The presence of oxygen and iron is considered, which could form iron oxides on the layers as a consequence of the corrosive action. As reported by Papadopoulos, Apostolopoulos, Alexopoulos and Pantelakis [25], the effect of iron oxide layers on corrosion is related to the blockage that limits the access of corrosive agents to the material. Moon et al. [23] found an irregular structure in the oxide layers produced on the steel, admitting the passage of corrosive elements and oxygen through the pores.

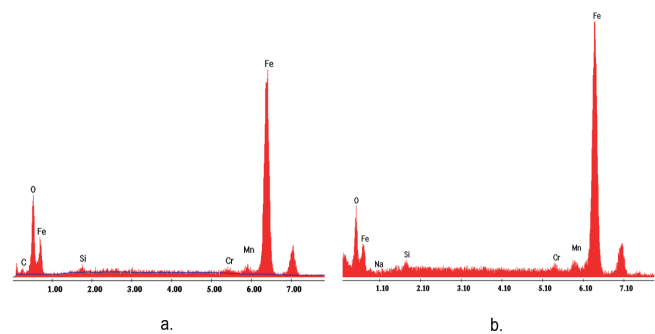


Figure 6. EDS spectra of the corroded surface: a. 40B; b. 60B

In the referred spectra the presence of chromium is observed, which has been object of analysis in this type of tests, as it is the case of the work developed by Qu et al. [9], where it is exposed that chromium contributes to the increase in the resistance to corrosion, due to the protective layer that is formed, and it is effective to increase the defensive influence of the oxides produced in steels. Likewise, Moon et al. [23] found in their analysis elements referring to chrome, copper, nickel and silicon, which contribute to the improvement in the protective capacity of the oxide layers.

Salt Water Test (Salt Chamber)

Salt chamber tests were developed to analyze the influence of boron on bainitic steels against corrosion resistance in saline environments, carried out as described in ASTM B117 [14].

The use of the chamber accelerates a corrosion process in a short time, because it leads qualitatively to a coastal

environment. Table 4 reports the results acquired by the test during a period of 24, 48, 72 and 100 hours, quantifying the resistance to corrosion through the percentage of corroded area generated on the surface of each steel.

Table IV. Percentage of area corroded.

Test tube	24 Hours (%)	48 Hours (%)	72 Hours (%)	100 Hours (%)	Standard deviation (%) (100 hours)
0B	87 ± 3,3	87 ± 3,3	87 ± 3,3	95 ± 2,9	5
20B	63 ± 26,7	68 ± 21,7	83 ± 16,7	92 ± 8	14
40B	90 ± 0	93 ± 3,3	97 ± 3,3	97 ± 3,3	5
60B	83 ± 5,8	87 ± 3,3	95 ± 1,7	95 ± 2,89	5

From the results described in the table, a very similar corrosion rate can be seen in steels, regardless of the boron content. For the first 24 hours there was a percentage of corroded area on the surface tested between 80% and 90%, except for steel 20B, whose surface was corroded by 63%. For the 48 hours the corrosion rate did not increase significantly on the surface of each steel. After 72 hours the percentage of corroded area increased in the 20B, 40B and 60B steels, while in the 0B steel its percentage remained constant. Finally, for the 100 hours, corresponding to the total time of the test, the average percentage of corroded surface reached 95%.

The above results indicate that among the steels there was no considerable difference between the corrosion rates at 100 hours, which may be associated to the fact that the percentage of ferroalloys used in each steel is similar, except for boron, allowing us to conclude that this element, for this type of test, does not influence the corrosion resistance of a bainitic steel.

Characterization of the corroded Surface

The micrographs in Figure 7 show the corrosion products on the surface of 20B, 40B and 60B steels, after 100 hours of exposure to the saline environment. It can be seen that the corrosion layer is not homogeneous on the surface, due to the presence of cracks on the oxide films, which is associated with the access of the chloride solution to the steel, promoting the corrosion process.

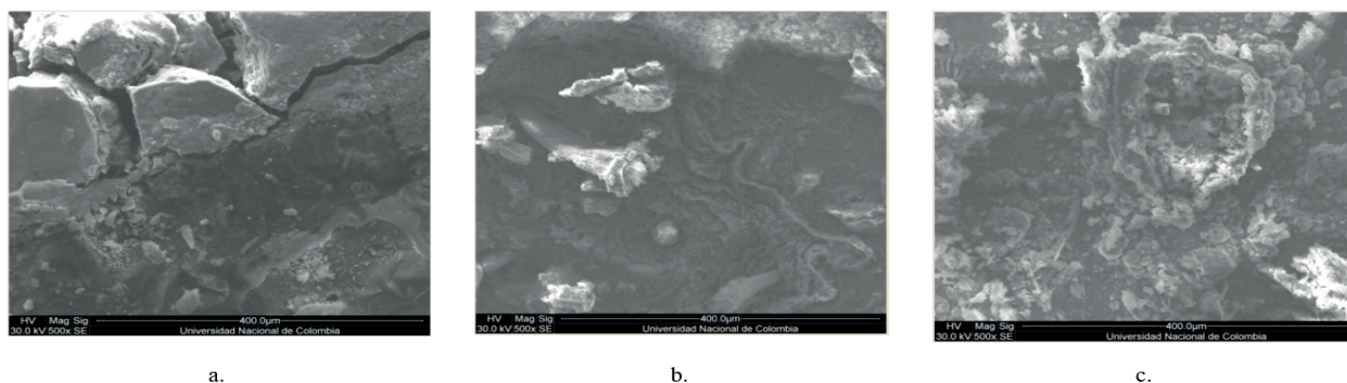


Figure 7. Corroded surface. a. 20B; b. 40B; c. 60B

On the other hand, in the micrographs small white products are observed, which can be related to sodium chloride compounds, which is associated if we take into account that in the EDS spectra (figure 8) obtained from the corroded surfaces, the presence of sodium and chlorine could be noticed, elements that are from the corrosive medium that was used for the development of the test.

The EDS spectra also show the presence of elements such as oxygen and iron, which are associated with the presence

of iron oxide on the corroded surface of each metal alloy. Additionally, alloying elements of the steel, such as silicon, manganese and chrome, are observed, which contribute to the densification and compactness of the oxide layers, thus reducing the diffusion of oxygen to the surface of the steel. Wang et al. [11] in their work related iron oxide (Fe_3O_4) as a corrosion product, associating chromium as an element of vital importance for the increase in the densification of the oxide layers, in order to reduce the oxygen diffusion to the surface of the material.

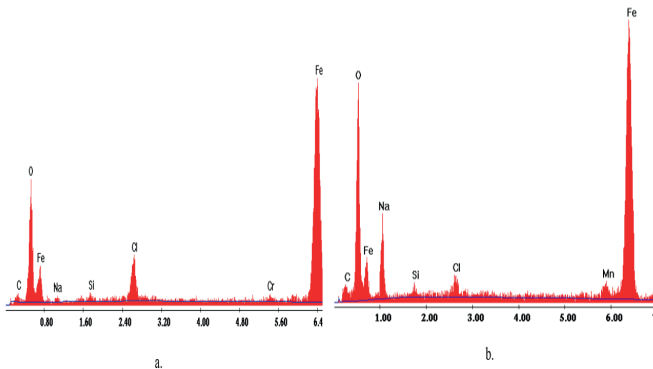


Figure 8. EDS spectra of the corroded surface: a. 40B; b. 60B

Conclusion

In the Tafel curves, obtained by means of the potentiodynamic polarisation test, a not very significant decrease in the corrosion speed was observed when the boron content increased, with the 60B steel having the lowest current density in the steels, around 23% lower density in relation to the steel that did not have boron. The results obtained by the test developed in a salt chamber showed a percentage of corroded area higher than 90% for all steels at the end of the 100 hours of test.

Corrosion products formed on the surface of the steels with the presence of oxides. In the salt chamber tests, elements of chlorine and sodium were found in the chemical composition of the corroded surface that came from the corrosive medium.

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