


Evaluating the Corrosion Behaviour of Borided Carbon Steel C35

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In this work, an evaluation of corrosion resistance of boride layers formed on carbon steel C35 in different solutions of H₂SO₄, HCl, HNO₃, NaCl and NaOH was carried out. Boriding treatment was applied on C35 carbon steel using Ekabor powder at 950 °C for 4 h. The corrosion behaviour was analyzed by the weight loss method after total immersion tests in different solutions at variable times from 24 hours to 6 months. The corrosion rate has been determined by electrochemical method using the polarization curves. Results obtained show that boriding treatment improved significantly the corrosion resistance of C35 steel in some acidic environments. The corrosion resistance of boride C35 steel is higher compared with that of untreated C35 steel, and the corrosion potentials E_{cor} of boride C35 steel are more electropositive than those of untreated C35 steel in some solutions.

Keywords: boriding, corrosion resistance, weight loss method, electrochemical method.

1. Introduction

Metal corrosion is a phenomenon of great economic importance. It limits its service life's and its structure, which leads to many replacement costs and loss of productivity, severely damaging many sectors of the industry. As all metals and their alloys generally corrode, corrosion in automobiles, microelectronic devices, various petroleum and chemical industries, boats, ship hulls, metals exposed to sea water, bridges, highways, and drainage channels is inevitable. The nature and solution of corrosion problems are closely related to the choice of materials. Corrosion tests can be performed in laboratories that have been enhanced by the same corrosion agents or in real-world contexts as faithfully as possible, and many of these tests are standardized. Gauges of weight loss, electrochemical resistance, and electrical resistance are among the specialized techniques for estimating corrosion of materials.

Corrosion cost studies are carried out in different countries, such as the USA, UK, Japan, Australia, Kuwait, Germany, Finland, Sweden, India, and China. They have shown that the economic penalties for metal corrosion are very serious. Corrosion in the oil and gas industry has been reported to be 25% of all safety accidents. Pipe systems are another major source of corrosion, leading to the largest environmental disaster in US history. Cleaning costs had surpassed 585 billion dollars and were expected to rise more than 20%. Conservative estimates of direct and indirect of corrosion costs are 6% of gross domestic product (GDP)¹. While the results of the studies show that the total estimated annual direct cost corrosion in 2001 was US \$ 276 billion per year, equivalent to 3.1% of the country's (GDP)^{2,3}. In their

study, Hou et al. (2017)⁴ estimated in their study that the cost of metal corrosion in China was roughly 2127.8 billion RMB ~ 310 and 14 342 billion USD in 2014 and 2019, respectively. Representing about 3.34% of the (GDP). It is generally agreed that 15– 35% of corrosion costs can be avoided via appropriate corrosion mitigation approaches, which implies that up to US \$ 118.16 billion of corrosion associated costs could be avoided annually in China alone. India loses up to \$ 100 billion annually due to corrosion. It influences a potential reduction in corrosion-related losses that could reach 5% of (GDP) in many countries.

There are several methods of protection against corrosion of metals; the application of shellac is an economical way to protect against corrosion. The coating acts as a barrier between the metal and the forcing agents. Thus, it prevents the flow of electrochemical charges between the two elements that corrode together. A negative measure of corrosion protection is the design of anti-corrosion structures, which increases the life and durability of all coatings. Ideally, structures should be designed in such a way that water and dust cannot penetrate to the metallic surface. The design should guarantee accessibility to the structure for periodic maintenance. Another way to protect against corrosion is to use corrosion inhibitors. These are chemicals that interact either with the surface of the metal or with the gases in the environment. The goal is to prevent the chemical reactions that occur that cause corrosion. Retarders and inhibitors are used to protect the metal surface. There are two ways to use chemical inhibitors. It can be mixed with a suitable solvent and then the resulting solution can be applied. On the other hand, it can be applied as a protective layer using common dispersion methods. Corrosion protection can be achieved by coating the metal surface.

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Thermochemical treatments are carried out to protect the metals and samples against corrosion. The surface treatments essentially aim at hardening the surface of the metal for better resistance to wear, friction, and corrosion. Boriding is a thermochemical surface treatment in which boron atoms are introduced through the surface of the treated material. The boron atoms react with the atoms of the base material by forming the boride layers with good properties like high surface hardness, good resistance to abrasive, and adhesive wear increased resistance to attack by acids and molten metals⁵⁻⁷. Temperatures process ranging between 850 and 1050 °C in either solid, liquid or gaseous, salt media. Boriding treatment is a prominent optimal for a wide range of tribological applications. It significantly increases the surface hardness and the wear resistance of metal. It consists of diffusing the boron across the surface of the metal substrate (steel, cast iron, titanium alloy...) ⁸⁻¹⁰. Since boron is a relatively small element, it diffuses into a variety of metals, including ferrous and most refractory alloys^{11,12}. Boron atoms can enter smoothly into the samples, forming a single or double phases of FeB and Fe₂B^{13,14}. The boride layer of ferrous alloys is generally composed of two sub-layers; the outer layer is FeB, while the inner layer is Fe₂B^{15,16}. The thickness and the proportion of each iron boride layer depend on the parameters used for this process (temperature and time), and amount of active in the environment, the temperature and the treatment time. Thus, the boride layer obtained is either single-phase (Fe₂B) or two-phase (FeB, and Fe₂B)^{17,18}. The thickness of the boride layer can reach 287 μm^{17,19}.

The main objective of this study was to evaluate the corrosion behaviour of boride C35 steel, in acid and basic solutions H₂SO₄, HCl, HNO₃, NaCl, and NaOH solutions. This work describes the various corrosion tests with weight loss method via total immersion tests at different times, and the electrochemical method through Tafel extrapolation and polarization curves.

2. Experimental Procedure

2.1. Sample preparation

The material that has been selected for this study is C35 carbon steel, whose chemical composition is given in Table 1 (expressed in weight percent).

The samples used were cut into a rectangular shape. For example, the use of samples of the same shape for all tests makes it possible to perform an equally valid comparative study. After cutting with a saw, the specimens were machined to the dimensions (10 x 12 mm²).

Samples preparation before testing generally involves two stages: the first one is a grinding operation using an emery paper, and the second one is a chemical cleaning with different solutions and reagents. Mechanical grinding is done with grade 120 SiC emery papers, 240 and 600 grit.

The industry standard specified for the surface finish corresponds to a finish on 600-emery paper. After grinding, the chemical preparation of the samples varies depending on the metals used. The procedures followed are those suggested by ASTM standards governing surface cleaning. All samples were ultrasonically cleaned with acetone, and then rinsed with distilled water before drying.

The boriding process was achieved at a temperature of 950 °C with an exposure time of 4 h, using Ekabor powder, composed of 5% B₄C, 90% SiC, and 5% KBF₄. Parameters are obtained from the results of the growth kinetics of the boride layers formed on the substrate^{17,19}.

2.2. Corrosion test

Corrosion tests of steel carried out in the laboratory are presented in the following order: firstly, calculate from the immersion tests. Secondly, those calculated from the data of potentiokinetic tests. Each immersion test comprises several periods of 24 hours until 6 months at ambient temperature. The majority of weight loss tests in the literature does not exceed about 4 days (100) hours²⁰, 7 days²¹, 9 days²², 10 days (240 hours)²³, 14 days, 20 days²⁴, and 33 days²⁵. However, this study achieved results up to 6 months (180 days). This method involves exposing samples to a corrosive medium to determine the time and measure the difference in mass of samples before and after each test. Samples are washed with distilled water, dried and weighed before and after each corrosion test. The only device that was used in this experimental part was a precise laboratory scale with a readability of 0.0001 g.

Electrochemical tests were performed with Voltalab (PGP201), the Potentiostat-Galvanostat associated with the Volta-Master 4 software allowed us to obtain the potentiokinetic curves of boride steel C35 treated in the same corrosive solutions (NaCl and H₂SO₄) used in the total immersion tests. In the second experiment, the study was limited to two solutions. The electrochemical measurements necessary to plot the curves of polarization are generally obtained with Potentiostat. This device is a direct current amplifier; it allows regulating the level voltage to a predetermined value by adjusting the current value. Considering that, the electrochemical corrosion tests made it possible to directly obtain significant data on the behaviour of the tested samples with respect to the corrosion medium taken into consideration. An electrochemical workstation was achieved with a conventional three-electrode electrochemical cell at room temperature. The platinum mesh counter electrode and the reference electrode, a saturated calomel electrode, were placed in the settled position. Polarization curves are obtained by the potentiokinetic method. Tafel curve is produced by the use of anodic and cathodic curves in different zones. Those curves made it possible to determine the corrosion potential E (I = 0) by the exploration of the Tafel lines and the determination of the corrosion current. This magnitude

Table 1. Chemical composition of C35 steel.

Element	C	Si	Mn	P	Cr	Ni	Fe
C35 steel	0.33%	<0.39%	0.60%	0.03%	0.30%	0.30%	Balance

has significance an importance for evaluating the corrosion rates from Faraday's law. These values of corrosion rates are provided automatically by the used software. For practical reasons, the corrosion rates will therefore be expressed in the usual unit, $\mu\text{m} / \text{year}$, by the application of Faraday's law and making the assumption that the observed corrosion is uniform²⁶.

2.3. Optical observation

Using an optical microscope, the boride layer and the substrate and corrosion attacks can be easily distinguished by the difference in color and contrast of the different phases. On the micrographs produced in this work, the boride layer is very illustrious. The cross-sections of the boride samples were experiential and examined by optical microscopy (Leica BM-600). Morphological and microstructural features were observed. The microstructural characterization of the etched specimens was examined by an optical microscope and prepared using standard metallographic techniques.

3. Results and Discussion

The corrosion behaviour was evaluated by weight loss curves after total immersion tests and by electrochemical methods. The treated samples were immersed in different corrosive solutions and their corrosion rates were compared to those of untreated samples immersed in the same solutions for the same periods.

3.1. Weight-loss method

The weight loss depending on the holding immersion time of samples with and without treatment in sulfuric acid (H_2SO_4) is shown in Figure 1. It should be noticed that there is a significant increase in corrosion rate at the beginning of the experiment, and after a few days, it becomes constant for the untreated sample. However, there was no considerable change in the weight loss. The lowest weight loss was observed for the boride C35 steel at $0.555 \times 10^{-3} \text{ mg/cm}^2$ at the first 24 hours, while the weight loss of the untreated specimen was 0.172 mg/cm^2 . This value increased to 9.557 mg/cm^2 after 1 month. The corrosion rate of the boride sample is negligible compared to that of untreated steel. Figure 1 shows clearly the difference between the

state of boride and untreated samples after 4 days in an aqueous solution of H_2SO_4 . The corrosion resistance of a boride sample is much higher than that of untreated steel. As shown in the corrosion graphic curves, the corrosion attack decreased after the application of boriding treatment. In addition, it was observed that the decreased solubility of corrosion in the boride specimens led to a decrease in their weight. The corrosion rate in the untreated specimen increased 8 times. This significant decrease in the corrosion rate and an increase in the corrosion resistance might be attributed to the formation of a protective layer. In the literature, the AISI 1050 steel exhibited significantly higher corrosion resistance in H_2SO_4 acidic media. It is obvious that the boriding treatment reduces the weight loss of the steel and considerably improves its corrosion behaviour²⁷.

In the case of HCl solution, the mass loss depends on the holding immersion time for C35 steel before and after boriding treatment is shown in Figure 2. The lowest weight loss observed for the boride C35 steel was 0 mg during the first 24 hours, while the weight loss of the untreated specimen was 16.580 mg/cm^2 . This value increased to 1098.238 mg/cm^2 after 1 month. As shown in the corrosion graphic curves, boriding treatment improves the corrosion resistance of HCl. The significant improvement that can be brought about by the boriding treatment is clearly visible. The corrosion of the boride sample is negligible compared to the untreated steel. Figure 2 also shows the difference between the state of boride and untreated samples after 4 days.

As exhibited in the corrosion graphic curves, the corrosion rate decreased with the boriding treatment. The corrosion rate in the untreated specimen increased 10 times. In addition, it was observed that the decrease in the corrosion rate of the boride specimens led to a decrease in weight loss. This significant decrease in the corrosion rate and an increase of the corrosion resistance can be ascribed to the formation of a protective boride layer.

The weight loss depending on the holding immersion time of C35 steel before and after boriding in HNO_3 is shown in Figure 3. In this case, the corrosion of the boride sample is negligible compared to the untreated steel. It is noticed that there is a significant increase in corrosion at the beginning of the experiment, and after a few days, it becomes constant for the untreated sample. The corrosion of

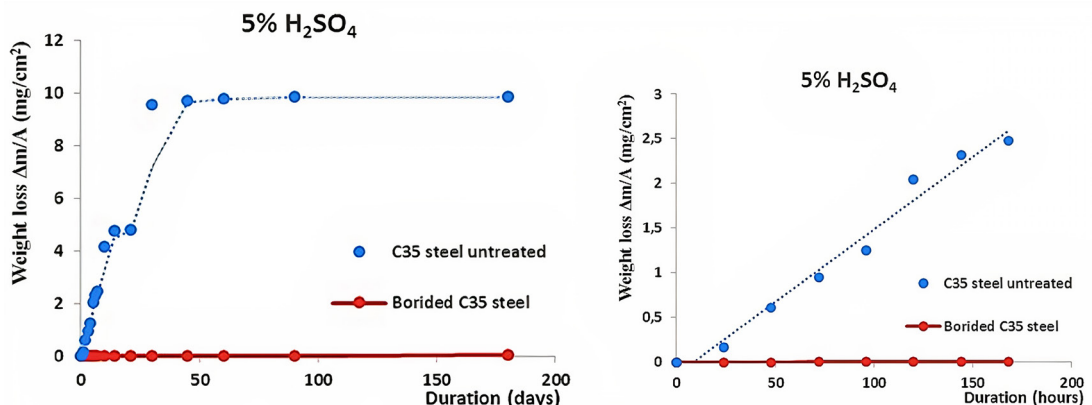


Figure 1. Corrosion rates depending on the immersion time of carbon steel C35 before and after boriding treatment in 5% H_2SO_4 solution.

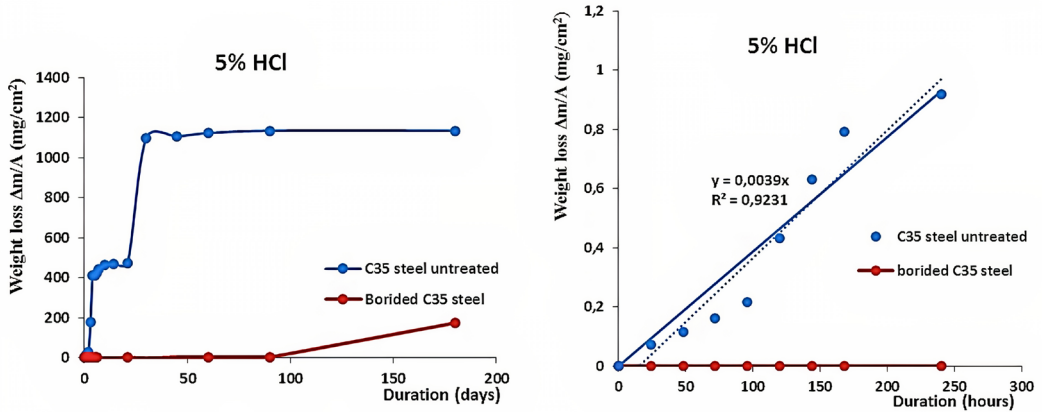


Figure 2. Corrosion rates depending on the immersion time of carbon steel C35 before and after boriding treatment in 5% HCl solution.

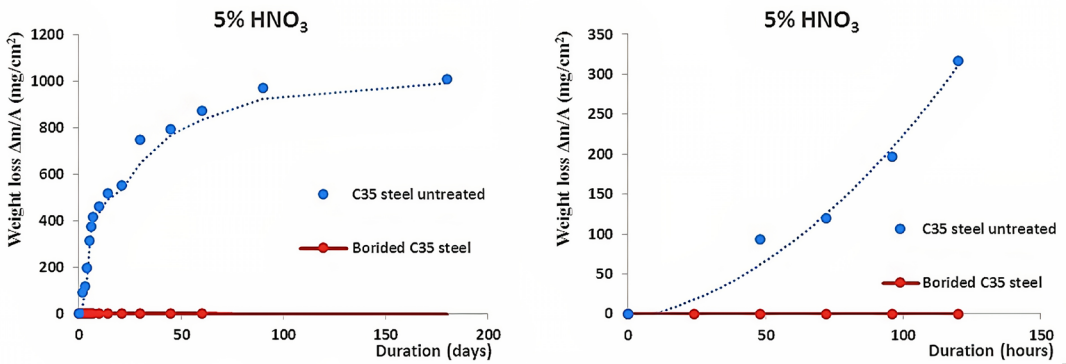


Figure 3. Corrosion rates depending on the immersion time of carbon steel C35 before and after boriding treatment in 5% HNO₃ solution.

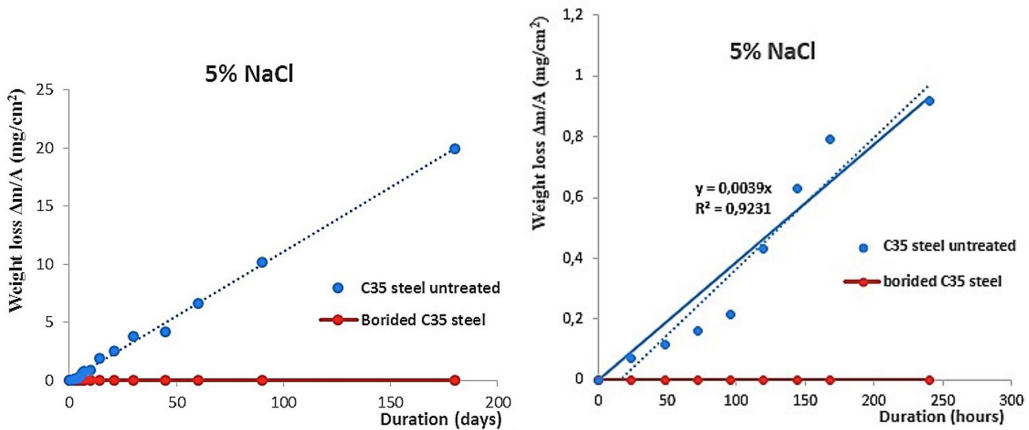


Figure 4. Corrosion rates depending on the immersion time of carbon steel C35 before and after boriding treatment in 5% NaCl solution.

C35 boride steel is negligible in comparison with untreated steel. Figure 3 depicts the difference in boride and untreated samples after a few days in an aqueous solution of HNO₃.

The lowest weight loss observed for the boride C35 steel was 0 mg for the first 24 hours, while the weight loss of the untreated specimen was 8.841 mg/cm². This value increased

to 1011.241 mg/cm² after 6 months. The corrosion of the boride sample is negligible compared to the untreated steel.

The weight loss depending on the holding immersion time of treated and untreated C35 steel in NaCl solution is shown in Figure 4. The corrosion rate of the treated specimen increased 14 times. It should be noted to constantly raised

corrosion rate of untreated C35 steel in the NaCl solution and the corrosion of the boride samples is negligible compared to the untreated steel.

The mass loss depending on the holding immersion time of C35 steel before and after boriding in NaOH solution is shown in Figure 5. There is significant corrosion and weight loss after 48 hours in the treated sample, but this corrosion stops after that, and it is stable and almost constant for six months. The lowest weight loss observed for boride C35 steel was 0 mg/cm² in the first 24 hours, while the weight loss of the untreated specimen was 0.162 mg/cm². This last value increased to 373.52 mg/cm² after 6 months. The corrosion rate of the untreated specimen increased 2 times after 21 days.

3.2. Corrosion observation

It can be seen clearly with the naked eye, the difference in behaviour between the boride steel and the untreated one in sulfuric acid can be seen clearly. The boride steel has practically kept its original shape and dimensions, while the untreated steel has completely lost its dimensions.

As shown in Figure 6, corrosion products were observed on the surface of the specimen after the corrosion test. The corrosion resistance of borided carbon steel usually depends on the characteristic features of coatings such as the number of micro cracks and porosities. These porosities negatively affect the firmness of coatings and significantly reduce the corrosion resistance. After the corrosion test, porosity and pits were not observed on the boride specimens. However, it was observed in un-boride specimens. These porosities negatively affect the firmness of coatings and significantly reduce the corrosion resistance. As a result of the boriding process, the corrosion resistance of C35 carbon steel increased compared to boride C35 carbon steel. In other part, the increase in the boride layer thickness affects both the surface roughness and corrosion resistance of C35 carbon steel. As the immersion time elapsed, the corrosion rate slowly or rapidly decreased according to the oxidation reaction of iron depending on the solutions. The corrosion rate showed a complicated pattern depending on the solution. Consequently, the changes in the corrosion rate and behaviour were closely related to the oxidation reaction of iron on the surface.

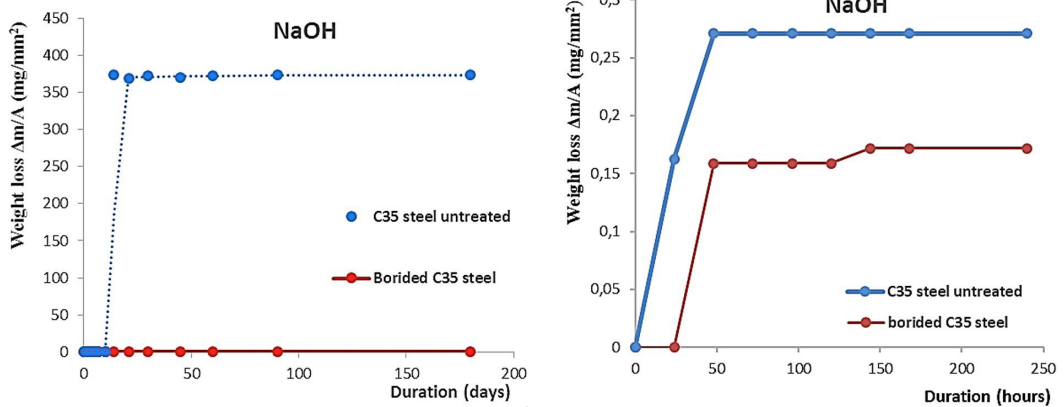


Figure 5. Corrosion rates depending on the immersion time of carbon steel C35 before and after boriding treatment in 5% NaOH solution.

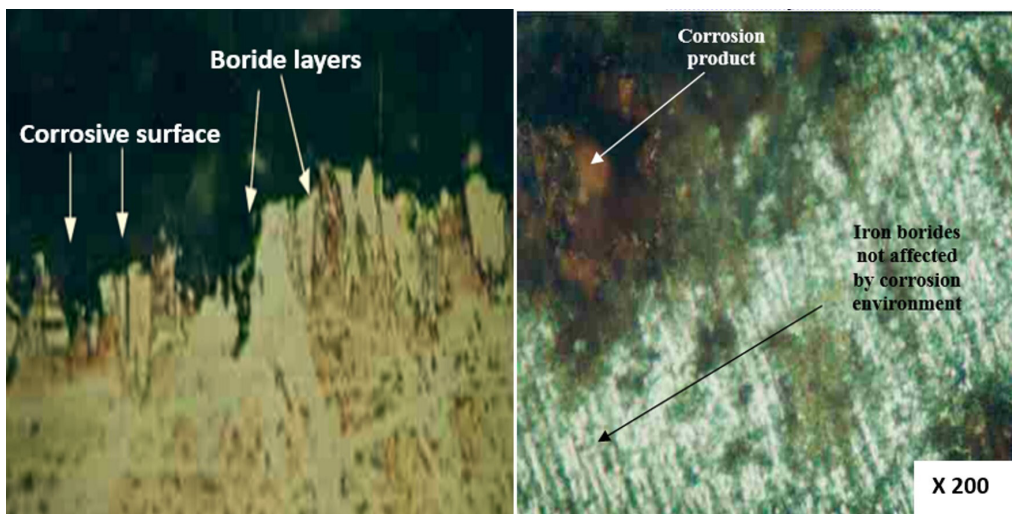


Figure 6. Corrosion on the surface of boride layers in 5% N₂SO₄ solution.

3.3. Tafel and polarization curves

Electrochemical parameters from polarization curves for different solutions are listed in Table 2.

Considering that the electrochemical corrosion tests make it possible to obtain directly significant data on the behaviour of the samples tested with respect to the corrosion medium taken into consideration, testes limited ourselves to the study of the boride samples without performing tests on the samples untreated.

The results of the Tafel extrapolation of the polarization curves for C35 carbon steel in NaCl solution are shown in Figure 7.

The corrosion resistance of the boride layer was investigated by the polarization curves. Figure 8 shows the electrochemical polarization curves (anodic and cathodic branches) for the untreated and boride C35 steel in 5% H_2SO_4 solution, respectively, at room temperature. It is seen that the values of corrosion potential E_{corr} of boride C35 steel are more electropositive than those of the un-borided state. This means that the boride layer can act as a protective barrier for the C35 carbon steel in this specific corrosion environment.

This result was confirmed and verified by the immersion test results, where the boride layer substantially reduced the weight loss in H_2SO_4 solution. A comparison of the samples appearance after immersion test gave a stronger impression that more than half of the plain sample had corroded after immersion for 4 days in H_2SO_4 solution. This result is similar to that reported in the literature^{25,28,29}. In fact, the boride AISI H13 steel showed a better corrosion behaviour in H_2SO_4 and H_3PO_4 environments, compared to untreated steel. On the contrary, the AISI H13 steel presented better corrosion resistance in HCl acid environment in comparison with the boride AISI H13 steel. The corrosion tests of C35 boride steel revealed that H_2SO_4 solution was more aggressive than NaCl solution. The corrosion resistance of the investigated steel in H_2SO_4 solution was increased by boriding. In addition, the corrosion resistance in NaCl solution was exacerbated by applying the boriding treatment. Compared to the results obtained in the literature, boride N80 steel showed an excellent corrosion resistance in both H_2SO_4 and HCl acid environments.

However, another study claimed that the boride AISI H13 steel did not reported a good corrosion resistance in HCl

Table 2. Electrochemical parameters from polarization curves.

Parameters	5% NaCl untreated	5% NaCl treated	5% H_2SO_4 untreated	5% H_2SO_4 treated
pH	7.642	8.010	1.150	1.07
E_{corr} ($I=0$) (mV)	-473.3	-438.1	-549.8	-425.2
β_a (mV/decade)	85.5	81.9	145.3	88.0
β_c (mV/decade)	-264.6	-315.3	-158.7	-305.1
I_{corr} ($mA\ cm^{-2}$)	0.1529	0.1887	0.2833	0.1467
Corrosion rate (mm/Y)	2.255	2.006	3.313	1.715

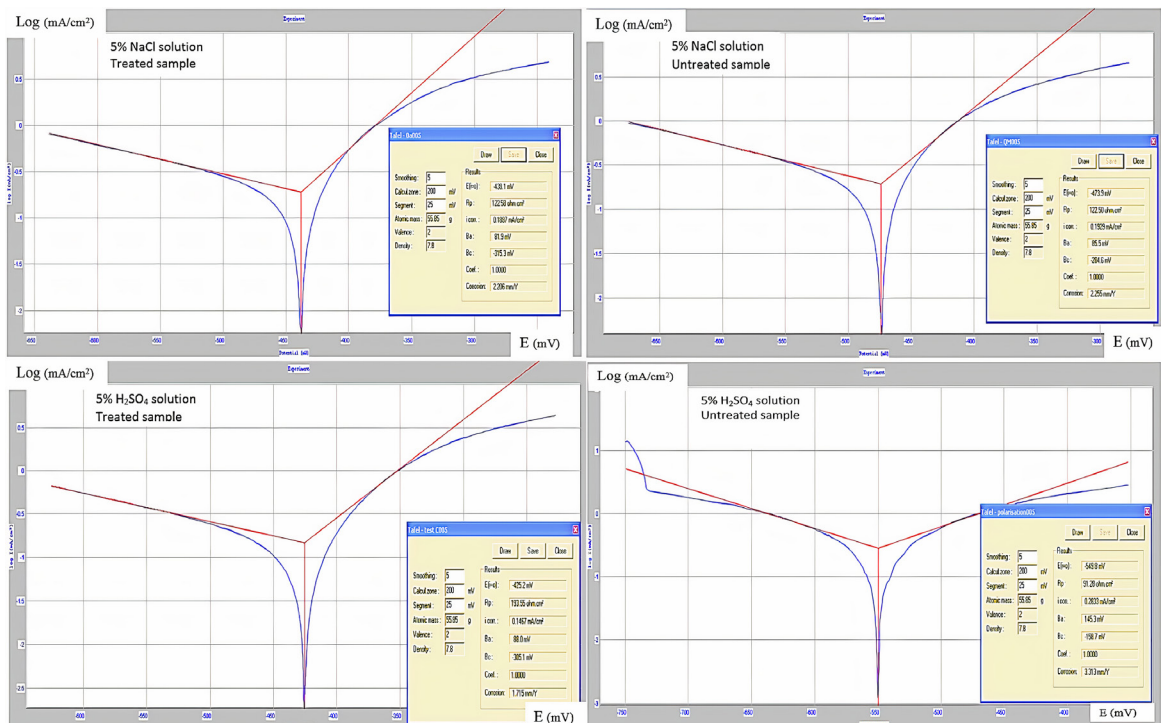


Figure 7. Tafel curves of treated and untreated C35 carbon steel in the solutions of 5% NaCl and 5% H_2SO_4 .

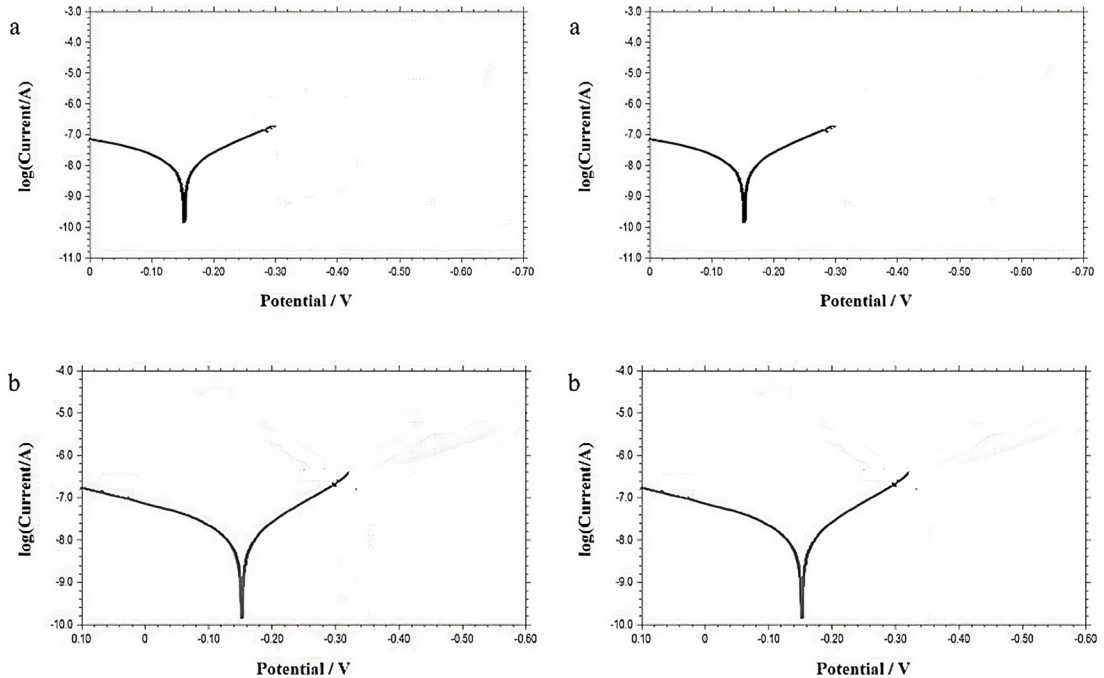


Figure 8. Polarization curves obtained from (a) Boride C35 carbon steel and (b) untreated C35 carbon steel.

acid environment²⁹. The corrosion behaviour and corrosion potential E_{cor} of C35 steel in H_2SO_4 solution were more electropositive than those of plain steels. Recent research suggests a higher corrosion resistance of the boride AISI H13 steel than the untreated one^{25,30}.

The results indicated that the corrosion resistance of C35 carbon steel increases when applying the boriding, and approaches the corrosion resistance of boride AISI 316L^{21,31}. The reported similar result showed that the boride layer considerably increased the corrosion resistance of the AISI 316L steel in HCl solution. While no positive effect of the boride layer was observed for NaOH and NaCl solutions, the corrosion resistance of the boride layer on C35 carbon steel was increased in all solutions with an increased immersion period. Thus, it can be confirmed that the boriding treatment improved the corrosion resistance of C35 carbon steel in the studied solutions.

4. Conclusions

Boriding treatment was applied on C35 carbon steel using Ekabor powder at 950 °C for 4 h. The samples were subjected to corrosion tests in several solutions (5% H_2SO_4 , 5% HCl, 5% HNO_3 , 5% NaCl, and 5% NaOH).

The corrosion resistance was expressed by the weight loss curves after total immersion, and by an electrochemical method. The corrosion rate of untreated C35 carbon steel in acidic environments in general, and especially in 5% H_2SO_4 , HCl, HNO_3 , and in aqueous NaCl, NaOH solutions is greater than the corrosion rate of boride C35 steel, as confirmed by an electrochemical testing.

Furthermore, the corrosion resistance of boride and non-boride C35 steels has been compared in this study. The boriding

treatment significantly improved the corrosion resistance of C35 steel. Thus, the corrosion resistance of borided C35 steel is higher compared with that of untreated C35 steel. By using the electrochemical polarization curves for the untreated and boride C35 steels in 5% H_2SO_4 solution, it was found that the corrosion potentials E_{cor} of boride C35 steel are more electropositive than those of untreated C35 steel. As a main concluding point, the corrosion resistance of boride C35 steel increased 4 to 16 times.

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