
PLASMA POWER IMPACT ON ELECTROCHEMICAL PERFORMANCE OF LOW CARBON STEEL COATED BY PLASMA THIN TEOS FILMS

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ABSTRACT

Electrochemical properties of thin silicon oxy carbide films were investigated as anticorrosive coatings. The film was deposited on low carbon steel substrate by radio frequency capacitive coupled plasma technique using tetraethyl ortho silicate (TEOS) as a precursor and Ar was used as a carrier gas in dependence on the applied power. The chemical composition and morphological of the deposited films were examined by energy-dispersive X-ray spectroscopy (EDX) coupled with scanning electron microscopy (SEM). The SEM results confirm a pinhole-free layer of oxy carbide was formed on the steel surface after plasma treatment.

The corrosion resistance of the coatings was analyzed by potentiodynamic polarization and electrochemical spectroscopy (EIS) in 3.5% NaCl solution at room temperature. The electrochemical results show remarkable corrosion resistance enhancement after plasma treatments. The corrosion current (i_{corr}) is significantly reduced from 12 $\mu\text{A}/\text{cm}^2$ for the blank sample to 1 and 0.3 $\mu\text{A}/\text{cm}^2$ for treated samples at 50, and 100 W, respectively. A marked increase of the protective properties was detected by 100 W sample with protective efficiency more than 98 % at room temperature.

Keywords: Corrosion resistance; Mild steel; Plasma treatments; Tetraethyl ortho silicate; Thin film

INTRODUCTION

Almost metals, particularly carbon steel, are thermodynamically unstable therefore, they corrode especially when they are in contact with oxygen and water. Moreover, the corrosion destroys millions of tons of carbon steel over the world per year [1,2]. Over the years, numerous numbers of studies that concerned with different methods for the protection of metals against corrosion to provide wide utilization of these metals. An effective way to retard corrosion of carbon steel by coating its surfaces with thin films which act as barriers against corrosion [3–7]. These films were applied to steel surface by different methodologies such as electrodeposition [8], hot dipping [9], sol-gel [8,9], chemical vapor deposition, and plasma-enhanced chemical vapor deposition (PECVD) [10–12]. However, the plasma-enhanced process is a promising technology for metals surface modification in terms of corrosion resistance enhancement [15]. This process is simple, economically feasible, and environmentally friendly.

The plasma deposited SiO_x thin films from organosilicon precursors manifest an attractive approach to enhancement of the corrosion resistance of carbon steel. The deposited SiO_x films showed excellent stability, flexibility, and smoothness [1].

Yung-Sen *et al.* [16] prepared protective coating on the steel surface by using a mixture of tetraethyl ortho silicate (TEOS) and H_2 at room temperature. They found that great improvement of electrochemical properties after immersion in 3.5% NaCl solution for 10 days. Rangel *et al.* [1] investigate the electrochemical properties of $\text{SiO}_x/\text{SiO}_x\text{C}_y\text{H}_z$ films in 3.5% NaCl solution too. The film was deposited by low-pressure radio frequency (13.56 MHz) plasmas using HMDSO, Ar and O_2 mixtures. It was noted that the total corrosion resistance increased by six orders of magnitude when carbon steel was coated with a 2 μm film. Delimi *et al.* [14] employed a mixture of SiH_4 (3% in N_2) and N_2O gas to deposit SiO_x coatings on carbon steel with a thickness between 20 and 200 nm. They

examined the corrosion protection efficiency in an aqueous saline solution 3% NaCl and report that thin SiO_x -like coating layers (20 nm thick) do not prevent the carbon steel from corrosion, while thicker silica layers ($d \geq 100$ nm) protect efficiently carbon steel interfaces in highly saline media with a protection efficiency of about 96% for a 200 nm thick coating. Saloum *et al.* [17] revealed the corrosion protection of plasma polymerized thin Hexamethyldisilane (HMDSO) films in 0.3 M NaCl and 0.3 M H_2SO_4 . A promising corrosion protection property of the thin HMDSO films was obtained. Ponton *et al.* [18] prepared thin and dense SiO_2 films from tetraethyl ortho silicate ($\text{Si}(\text{OC}_2\text{H}_5)_4$, TEOS) using chemical vapor deposition (CVD) in the range of temperature from 400 to 550 °C with effective electrochemical properties.

In summary, the organic silicon compounds like TEOS may be the right choice for the deposition of good and uniform thin films of silicon oxide (SiO_2), which may provide good protection for carbon steel against corrosion.

This work aims to prepare protective coatings on carbon steel by applying TEOS as a precursor plasma technique based on plasma power. Furthermore, topography, elemental analysis, and the electrochemical properties of the protective films were examined.

2. MATERIALS AND EXPERIMENTAL METHODS

2.1. Materials

Mild carbon steel substrates obtained from Central Metallurgical Research Institute, with composition of (in wt%) 0.271 % C, 0.214 % Si, 0.829 % Mn, 0.0305 % P, 0.0156 % S, 0.0122 % Cr, 0.0187 % Ni, 0.00332 % Al, 0.0041 % Co, 0.0071 % Cu, 0.00014 % Ti, 0.00275 % V, 0.00951 % W, 0.00951 % Pb, 0.0108 % As, 0.0003 % B and 98.57 % Fe was used in this study.

For all the experiments, the cylindrical carbon steel samples with dimensions (25 mm in diameter and 5 mm in height) were polished with silicon carbide emery papers with different grades (60, 80, 120, 400, 800, 1000 and 2000) to produce a fine surface. After that, the

samples were washed with acetone and dried at room temperature before use.

Tetraethyl orthosilicate (TEOS) $\text{Si}(\text{OC}_2\text{H}_5)_4$ with molar mass 208 g/mol was obtained from Sigma-Aldrich analytical grade 99 %.

2.2. Corrosive solutions

Sodium chloride (NaCl) with analytical grade 99.99% was used to prepare a 3.5 % saline solution in deionized water for electrochemical characterization.

2.3. Plasma deposition method

The plasma discharge was generated in a stainless-steel reactor chamber using radio frequency plasma produced by a power supply (RFG-1K-13, UK) with 13.6 MHz and continuous out power ranging from 0 to 1000W. The schematic representation of the experimental set-up is shown in Fig. 1. The steel substrates were then placed in the plasma chamber as electrodes at room temperature. Subsequently, the chamber was pumped down to 300 m Torr using a vacuum pump. Thereafter, the Ar gas was introduced into the bubbler system containing (TEOS) liquid and carry the TEOS vapor into the plasma chamber. After the pressure became stable at 1 Torr, the power supply was switched on for 10 minutes. This experiment was carried out with a wide range of plasma power (50, 100, 150, 200 and 250W).

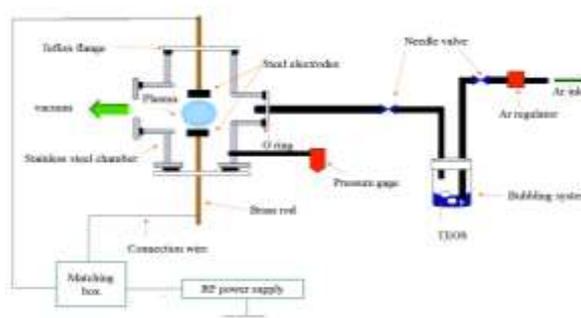


Figure 1. The schematic diagram of the plasma deposition cell.

2.4. Characterization methods

2.4.1. Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX)

The surface morphology of samples was examined using SEM (FEI-inspect -S50,

Denmark) with magnification rang to 100,000 x. SEM is attached with an EDX unit with acceleration voltage 30 KV and used for elemental analysis of the treated sample compared to the untreated ones.

2.4.2. Electrochemical measurements

Electrochemical investigations were carried out using a Potentiostat / Galvanostat / FRA analyzer (Auto lap PGSTAT 30, Netherlands), in the classical three-electrode cell containing saturated calomel reference electrode (SCE) and a platinum grid as a counter electrode. Moreover, the surface area of the working electrode was 1 cm².

Two electrochemical techniques were used for the evaluation of corrosion protection efficiency of treated samples, one of them is electrochemical impedance spectroscopy (EIS). The impedance measurements were carried out by applying an AC excitation of 10 mV amplitude (peak-to-zero) of sinusoidal voltage in the frequency range from 100 kHz to 10 m Hz. The samples were immersed in a 3.5% NaCl aqueous solution for 30 minutes to acquire a steady state before measurements. All measurements were completed at room temperature (30°C).

The obtained impedance data were analyzed using Z view 2 software to achieve the best fitting and calculate EIS associated parameters. The inhibition efficiency (η_{EIS}) was calculated using Eq. (1).

$$(\eta_{EIS}) = [1 - (R_{ct}^{\circ} / R_{ct})] \times 100 \quad (1)$$

Where R_{ct}° and R_{ct} are the charge transfer resistance of untreated and treated steel samples, respectively.

Potentiodynamic polarization is also used to evaluate corrosion performance and was performed by scanning applied potential from -0.3 to 1 mV at a scan rate 2 mV s⁻¹. The inhibition efficiency (η_{pol}) was calculated from Eq. (2).

$$(\eta_{pol}) = [1 - (I_{corr} / i_{corr}^{\circ}) \times 100] \quad (2)$$

Where i_{corr}° and i_{corr} are the corrosion current densities of untreated and treated samples, respectively.

3. RESULTS AND DISCUSSION

3.1. Surface morphology

Surface topography was investigated using SEM. Fig. 2 exhibits the morphological characteristics of treated and untreated steel samples before and after immersion in 3.5% NaCl solutions for 24 h with power inlet 100 and 250 W. It is seen that the untreated steel sample before immersion was smooth and pits free (Fig.2a). The treated sample at 100 W exhibit that the steel surface is covered with a smooth, dense, and homogeneous film, as seen in Fig 2b. Conversely, the film obtained with 250 W was non-uniform. It contains some cracks and gaps as shown in Fig.2c. This observation indicates the film morphology strongly depends on the plasma power. Indeed, the raising of power creates high energized ions that allow to sustains reactions in the gas phase and little bet of materials were deposited at the film [19]. Also, the energized ions may collide with the film which previously deposited causing ruptures and cracks upon it [20].

Fig. 2d reveals the surface morphology of the untreated sample (blank) after dipped in the corrosive solution. The untreated surface appears damaged and pitted, because of chloride ions attack on the steel surface. The plasma-treated sample at 100 W seen be protected and little affected by the corrosion medium as presented in Fig. 2e. While a large damage area of the film was obtained with 250 W (Fig. 2f), that might be related to the fragmentation of the monomer followed by poly-recombination due to the high energy of plasma leading to a poor quality deposited film (irregular structure) [21, 22]

3.2. Elemental analysis using energy dispersive analysis of X-ray (EDX)

EDX spectrum analysis provides a clear understanding of the elemental composition of the steel surface. The obtained results were revealed in Table 1. Silicon, oxygen and carbon were detected in all treated samples. Moreover, the atomic percentages (At %) of previous elements follow a regular trend based on plasma power.

The highest Si, O, and C; and lowest Fe contents were observed in the treated sample

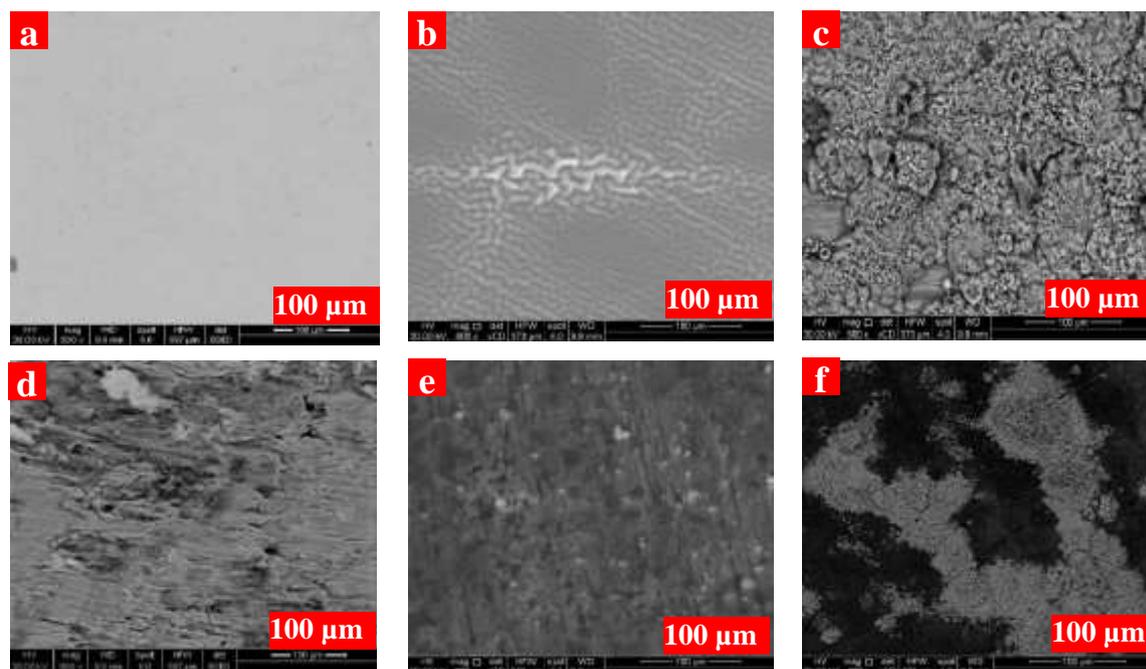


Figure 2. SEM image of carbon steel sample as: blank (untreated) (a), treated sample with 100 W (b), 250 W (c); where these samples after 24-hour immersion in 3.5% NaCl solution: blank (d), 100 W (e), 250 W (f).

with power 100 W. On the other hand, the prepared sample with 250 W, the atomic percentage of Si and O are low while the carbon content is undetectable compared to that sample which made with 100 W.

Theoretically, the atomic percent should be in the TEOS as: 07.7 Si, 30.8 O and 61.5 C.

The data obtained confirm that an irregular film was obtained with high power inlet than 100 W.

From this data, it might be proposed that the composition of the deposited layer is silicon oxy carbide like matrix.

Table 1. Elements composition (atomic %) of deposited films with different power compared to the blank sample obtained by EDX analysis.

Plasma power (W)	Element (At%)			
	Fe	Si	O	C
blank	99.36	—	—	00.64
50	76.46	2.75	4.63	16.16
100	76.80	6.73	5.67	10.80
150	90.33	2.54	2.35	04.78
200	93.62	1.49	2.13	02.76
250	98.02	0.84	1.14	—

3.3. Electrochemical investigations

3.3.1. Potentiodynamic polarization measurements

The potentiodynamic polarization curves for carbon steel with and without the plasma treatment in 3.5% NaCl at 30 °C are presented in Figure 3. The electrochemical parameters extracted from this figure such as corrosion potential (E_{corr}), corrosion current density (I_{corr}), corrosion rate (K_{corr}), corrosion inhibition efficiency ($\eta_{pol}\%$), anodic and cathodic Tafel slope (β_a , β_c), are given in Table 2. It was noted that all polarization curves shift to lower current densities by plasma treatment. However, the depletion in current density is directly proportional to the power of plasma and reaching a minimum at 100 W of power. The reduction of I_{corr} indicates that the deposited thin film of plasma TEOS has an ability to protect the carbon steel in the corrosive solution with protective efficiency 97.29 and 90.58% for 100 and 50 W, respectively. However, when the power was reduced to 50 W the energy of ions in the plasma sheath is not satisfactory enough to decompose TEOS in the gas phase which minimizes the deposition rate of film.

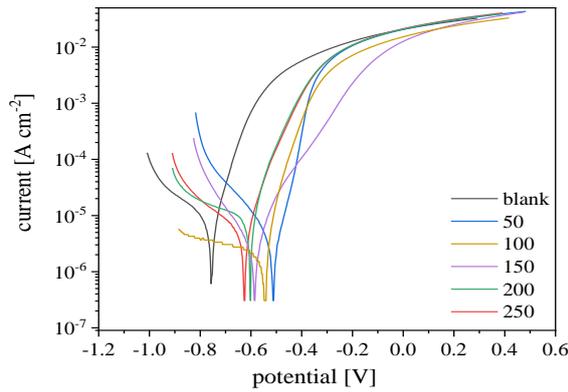


Figure 3. Potentiodynamic polarization curves of untreated and plasma-treated steel with different plasma power in 3.5% NaCl at room temperature (30°).

The values of E_{corr} for the treated steel is higher than that obtained for blank. The corrosion potentials (E_{corr}) values for the treated samples in a 3.5 % NaCl solution are increased with increasing the plasma power as shown in Table 2. The increase in E_{corr} and decrease in I_{corr} under plasma treatment and reach to the optimum at plasma power (100W) which proved to be effective in protecting the substrate from the corrosive medium.

3.3.2. Electrochemical impedance spectroscopy (EIS)

The effect of plasma on the corrosion behavior of mild steel in 3.5% NaCl was also investigated by electrochemical impedance spectroscopy. The EIS data are given as Nyquist and Bode-phase angle plots (Figs.4 and 5), respectively, in 3.5% NaCl at 30 °C. The Nyquist plot shows semicircular hoop over the entire frequency range which may be attributed to the occurrence of the charge transfer process in the solution which controls the corrosion mechanism of steel [13].

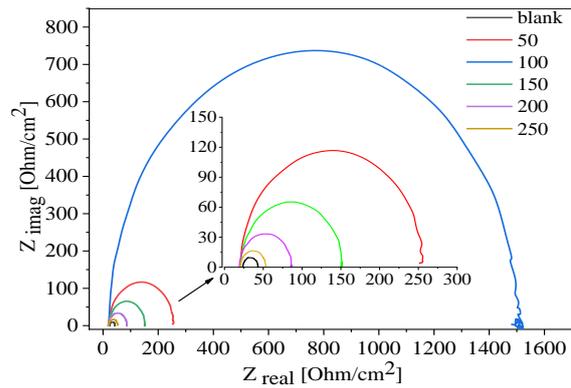


Figure 4. Nyquist plot of base material and plasma-treated samples in 3.5% NaCl at room temperature(30°).

Moreover, the EIS parameters such as double-layer capacitance (C_{dl}), charge transfer resistance (R_{ct}), and efficiency of inhibition ($\eta_{EIS} \%$) were collected in Table 3. The charge transfer resistance of carbon steel was increased significantly by plasma treatments, and the enhancement degree was increased to maximum as the power of plasma reached to (100W). Indeed, as mentioned before, the increase of plasma power creates highly energetic ions in the gas phase which prevents the deposition of a uniform protective film over the steel surface [23]. Moreover, this hypothesis is good agreement with data obtained by SEM that mentioned before. The double-layer capacitance (C_{dl}) is used to display the capacitance of a double layer formed at the interface between the steel surface and the electrolyte [24]. Therefore, that decreased after plasma deposition indicating to hydrophobic nature of the deposited layer that's slowing down the interaction between the electrolyte and coated steel providing good protection against corrosion.

Table 2. Potentiodynamic polarization associated parameters of low carbon steel coated by plasma deposited thin films at various plasma power in an aqueous solution of 3.5% NaCl at room temperature.

Plasma power (W)	i_{corr} ($\mu A/cm^2$)	E_{corr} vs. SCE(mV)	B_a (mV.dec ⁻¹)	B_c (mV.dec ⁻¹)	K (mm/year)	η_{pol} (%)
blank	12.001	- 0.8093	115	-203	0.08981	—
050	01.130	-0.5144	171	- 134	0.00845	90.58
100	00.325	- 0.5410	025	- 222	0.00243	97.29
150	01.900	-0.5872	072	- 081	0.01422	84.16
200	03.552	-0.6052	173	- 066	0.02658	70.40
250	06.932	-0.6540	056	- 063	0.05188	42.23

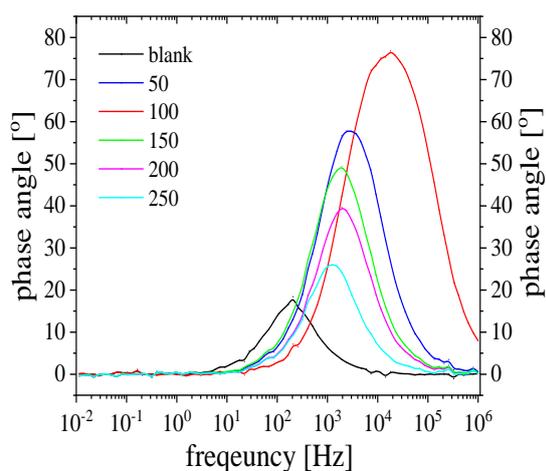


Figure 5. Bode plot of untreated and plasma-treated samples in 3.5% NaCl at room temperature (30°C).

Simultaneously, in the Bode-phase diagram the carbon steel in corrosive solution exhibit one peak at the middle frequency range, which refers to the changes in the phase angle caused by substrate-electrolyte interactions (Figure 5). This peak is still detected after plasma deposition but shifted to high frequency region and gets more intense in all treated samples. The simultaneous shift of the phase angle to high frequency region is because of coating/electrolyte interface. Also, this shift was reaching the maximum in the deposited sample with 100 W. This behavior might be related to the better adhesion of the coating and reveal a better performance of the sample in the corrosive medium.

The obtained EIS data were analyzed using an electrical equivalent circuit to achieve the best fitting of these data. The Randle circuit (Fig. 6) consists of two resistances and capacitance. R_s is the solution resistance between the reference and the working electrodes, R_{ct} represents the charge-transfer resistance at the metal/electrolyte interface. The capacitance C_{dl} signifies the interface between the working electrode and the corrosive electrolyte.

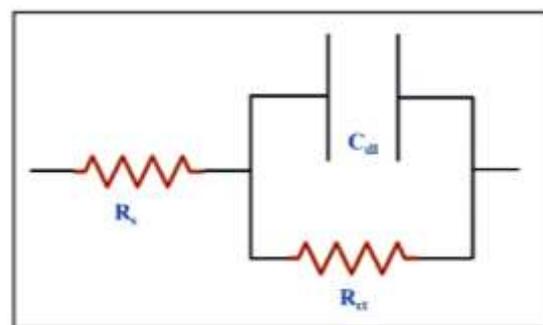


Figure 6. The electrical equivalent circuit (Randle equivalent circuit model) used to fit EIS data.

Table 3. EIS parameters of low carbon steel coated by plasma deposited thin SiO_xC_y films with different power in the aqueous solution of 3.5% NaCl at room temperature.

Plasma power (W)	R_s ($\Omega \text{ cm}^2$)	R_{ct} ($\Omega \text{ cm}^2$)	C_{dl} ($\mu\text{F cm}^{-2}$)	η_{EIS} %
blank	23.80	0019.37	56.924	—
50	20.79	0235.20	00.819	91.76
100	22.46	1485.00	00.0517	98.69
150	21.20	0130.50	01.800	85.15
200	19.34	0066.98	02.500	71.08
250	20.60	0032.80	06.280	40.94

CONCLUSIONS

Silicon oxy carbide thin films were deposited successfully on the steel substrate by RF/TEOS plasma at room temperature. The structure/property relationships of treated samples were studied in dependence on the plasma power. The deposited films exhibit a uniform surface without apparent pinholes. Polarization and electrochemical impedance spectroscopy analysis indicates that thin TEOS films act as a barrier layer between metal/solution interfaces that enhances the corrosion resistance of carbon steel. The deposited film which prepared with 100 W is the best condition with protective efficiency more than 98 % at room temperature. Moreover, these films can provide good anti-corrosion property with a cost-effective approach.

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تأثير قوة البلازما على الأداء الكهروكيميائي للصلب منخفض الكربون المطلبي بأغشية البلازما TEOS

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الملخص العربي

تم دراسة الخصائص الكهروكيميائية لعينات الصلب الكربوني المطلبي بأغشية أوكسي كربيد السيليكون وذلك من أجل تقييمها كطلائحات مضادة للتآكل. تم ترسيب هذه الأغشية على عينات الصلب منخفض الكربون بواسطة تقنية البلازما وذلك باستخدام مخلوط غازي مكون من غاز الأرجون ورباعي إيثوكسيد السليكون. تم فحص التركيب الكيميائي للأغشية المُرسبة بواسطة التحليل الطيفي للأشعة السينية (EDX) إلى جانب الفحص باستخدام المجهر الإلكتروني (SEM). وتشير نتائج المجهر الإلكتروني إلي تكون طبقة متجانسة وخالية من الثقوب من أوكسي كربيد السيليكون على سطح الصلب بعد معالجة البلازما. أيضا تم إختبار مقاومة التآكل لهذه الطلاءات بواسطة تقنيتي التحليل الطيفي الكهروكيميائي وإستقطاب الجهد الديناميكي في محلول كلوريد الصوديوم بتركيز 3.5% عند درجة حرارة الغرفة 30 °م. وأظهرت نتائج الإختبارات الكهروكيميائية تحسناً ملحوظاً في مقاومة التآكل للصلب الكربوني بعد معالجة البلازما لتصل نسبة الحماية من التآكل إلي أكثر من 98% للعينات المعالجة عند تطبيق طاقة قدرها 100 وات للبلازما في درجة حرارة الغرفة.