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A Case Study: Investigation of Untreated and Treated 304 Stainless Steel on Corrosion Behaviour

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ABSTRACT

A titanium coating on an iron-based metal surface significantly enhanced its resistance to localised corrosion. The research thoroughly investigated the microstructure and corrosion behaviour of both the untreated and treated 304 stainless steel substrates. The coating's morphology was meticulously examined using scanning electron microscopy (SEM), while its chemical composition was determined via energy-dispersive X-ray spectroscopy (EDX). Electrochemical impedance spectroscopy (EIS) was employed in an open circuit potential experiment to evaluate the coating's resistance to localised corrosion in an alkaline solution. SEM was again utilised to assess the coating's morphologies and cross-sectional view. The result revealed that untreated samples showed small and large pits on the microstructure, while no pit was detected in treated samples. Only fine dimples and voids were observed for the treated sample. The treated sample exhibited superior corrosion resistance to the untreated sample with a corrosion rate of 0.002348 mm/year and 0.007109 mm/year, respectively. This is attributed to the presence of the coating for a treated sample with curing for 10 minutes. The corrosion rate value is still considered excellent and accepted for stainless steel because the corrosion rate penetration is below 1 mils per year (mpy).

1. Introduction

Austenitic stainless steels such as 304 stainless steel (SS304) are widely used in industrial fields because of their excellent mechanical properties [1-3], high corrosion resistance especially to localised corrosion [1,3], good chemical resistance [1], and high formability [3]. The use of stainless steel has attracted considerable interest. It has a very thin protecting, passivating, self-renewing, and self-repairing layer on the surface. Even though stainless steel alloys resist corrosion, they may be exposed to external influences such as halide (Cl-) ions, pH fluctuations, and temperature variations over time. All of these factors stimulate the corrosion phenomenon [4].

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Nowadays, commercially advantageous protective technologies confront new obstacles, and various measures are being considered, including protective coatings applications. Based on the partial reaction (anodic and cathodic), the reaction mechanism (adsorption, passivation, film precipitation, and corrosive agent elimination), and the field of application (alkaline medium, acid medium, neutral medium, paints, gaseous phases), corrosion inhibitors are categorised [4]. For instance, applying metallic coatings on stainless steel surfaces is considered one of the most efficient, practical, and cost-effective methods for safeguarding metals and alloys in varied corrosive environments. Moreover, it is also functional when it reacts with aggressive substances such as alkaline and salt solutions [2,5]. These coatings create a protective barrier that improves the resilience of alloys against environmental variables that lead to corrosion and wear. These procedures are specifically developed to establish the obstacles that shield metal surfaces from detrimental ecological factors, prolonging the material's durability [6].

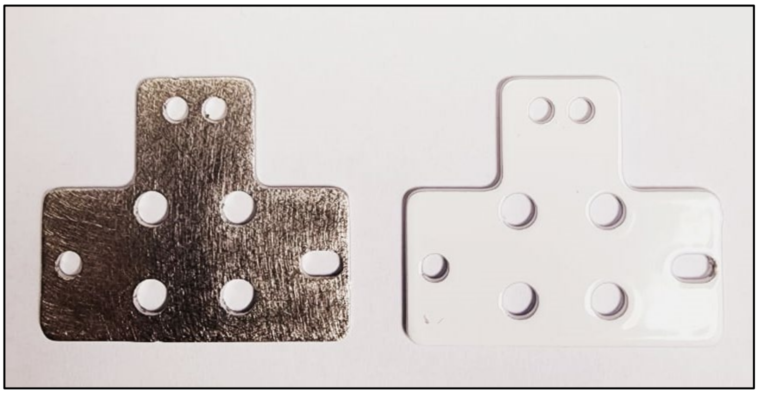
Nevertheless, stainless steel is susceptible to corrosion, particularly in alkaline settings, which can gradually undermine its structural integrity. Corrosion in alkaline settings can have a severe negative impact on stainless steel, causing the formation of pits and deterioration of the material [7]. There are five commonly utilised chemical coatings for stainless steel including titanium oxide/butoxide coatings [8], titanium nitride and oxynitride coatings [9], nanostructured sol-gel TiO_2 films [10], Cr or Ti co-doped amorphous carbon films [11] and titanium-silicon-carbon (Ti-Si-C) films [12]. Each coating functions through distinct methods to safeguard stainless steel from corrosion. However, titanium oxide (TiO_2) coatings effectively decrease corrosion rates by creating a barrier [8]. Ceramic oxide films such as TiO_2 coatings can be deposited on metals to improve their surface properties due to their excellent passivity, low conductivity or insulating properties and good tribological properties, showing good corrosion resistance in aggressive media [10,13-16].

Despite stainless steel's inherent corrosion resistance, corrosion incidents during production persist. The precise mechanisms by which smooth powder-coated surfaces (treated surface) impact corrosion resistance remain inadequately elucidated. Even though powder coating offers numerous advantages, its efficacy on stainless steel surfaces hinges upon meticulous preparation, precise application techniques, and stringent quality control measures throughout production. However, corrosion still happens during production, making the part rejection too high. Therefore, this study aims to investigate untreated and treated 304 stainless steel surfaces and their implications for corrosion resistance via electrochemical impedance spectroscopy.

2. Methodology

2.1 Sample Preparation

Both samples of 304 stainless steel are shown in Figure 1. The dimension of the samples was 33x39x1 mm. During sample preparation, the sample was polished to ensure the surface was smooth and free from scratches and burrs by using a portable power tool sanding machine model 3M™ Elite Random Orbital Sander. Then, the sample was treated using chemicals type-A during phosphating process. In this process, a metallic surface reacts with a phosphate solution, forming a tough, insoluble layer of metal phosphates. This process starts with pre-cleaning the material with an acid solution before applying the phosphate layer. The resulting metal phosphate layers, ranging from pale to dark grey, are firmly anchored in the metal surface, containing many cavities and capillaries. This unique structure allows the phosphate layers to absorb oils, waxes, colour pigments, and lacquers effectively, making them valuable for corrosion protection and as a base for paint and varnishes. After that, titanium-based (Ti-based) phosphate liquids are applied to the substrate surface using the spray or powder coating technique. The sample was then exposed to the curing process in the oven at temperatures 163 to 204 Celsius (°C) for 10 minutes.



(a) (b)

Fig. 1. The 304 stainless steel (a) Untreated (b) Treated surface

In order to facilitate cross-sectional examination, an adhesive coating is applied for a cold mounting process as represented in Figure 2. This preserves and maintains the coated substrate during the corrosion test. The mixture of resin and hardener was stirred thoroughly to remove bubbles and ensure uniformity. Applying a generous amount of releasing agent into the mounting cup facilitated the removal of the cured epoxy. The epoxy mixture was poured into the mounting cup, and the sample was securely attached. The mounted sample hardened over 24 hours.

Fig. 2. Cold-mounted samples

Corrosion testing was conducted using the Autolab PGSTAT-30 GPES system. The electrochemical cell configuration included a working electrode (sample), a Mercury/Mercury Oxide (Hg/HgO) as reference electrode, and a Platinum (Pt) as wire counter electrode, which was tabulated in Table 1. The setup utilised a 6M potassium hydroxide (KOH) as an electrolyte solution. The (Hg/HgO) electrode is chosen for its stability and reliability in alkaline environments, offering a wide potential range and a reversible redox process for accurate electrochemical evaluations. The platinum wire electrode, known for its lack of chemical reactivity and durability, ensures reliable electrochemical reactions for precise corrosion analysis in alkaline solutions. Electrochemical Impedance Spectroscopy (EIS) conducted at the open circuit potential (OCP) allows for the assessment of the corrosion rate and corrosion mechanism of iron (Fe) in alkaline conditions. The impedance spectra are acquired from EIS tests to evaluate the corrosion mechanisms, detect the existence of corrosion byproducts, and measure the efficiency of corrosion prevention techniques [17]. All parameters of the experiments are shown in Table 2.

Table 1
Electrode profile

Electrode	Description
Hg/HgO	Reference electrode
Platinum	Counter electrode
Copper wire (sample)	Working electrode

Table 2
Open circuit potential (OCP) parameters and properties

Parameter	Range
Start potential	$-0.5\ V_{ocp}$
Stop potential	$0.5\ V_{ocp}$
Scan rate	0.01 V/s
Step	0.001
Automatic current ranging (highest)	1 mA
Automatic current ranging (lowest)	10µA
Surface area	$26\ cm^2$

A single copper wire is coiled and attached to the sample, as illustrated in Figure 3. During the experiment setup, the continuity mode on a multimeter is utilised and checked to ensure that the circuit is closed on the substrate. Then, when the wire has no resistance, allowing power to flow freely, the multimeter will pass a small current and emit a beep. However, the circuit or wire must be replaced if there is no sound or the multimeter reads an open loop (OL). The experiment can be started when the setup is ready.

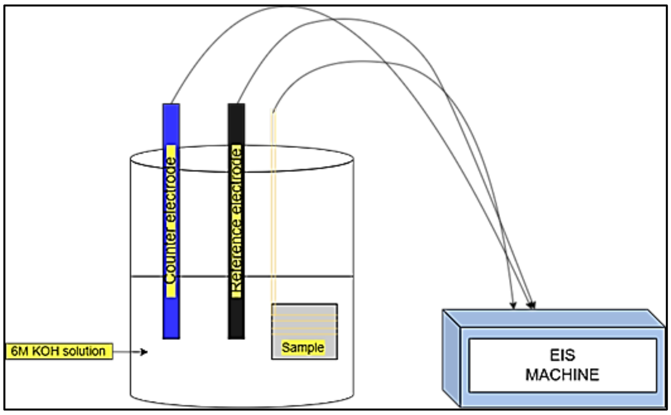
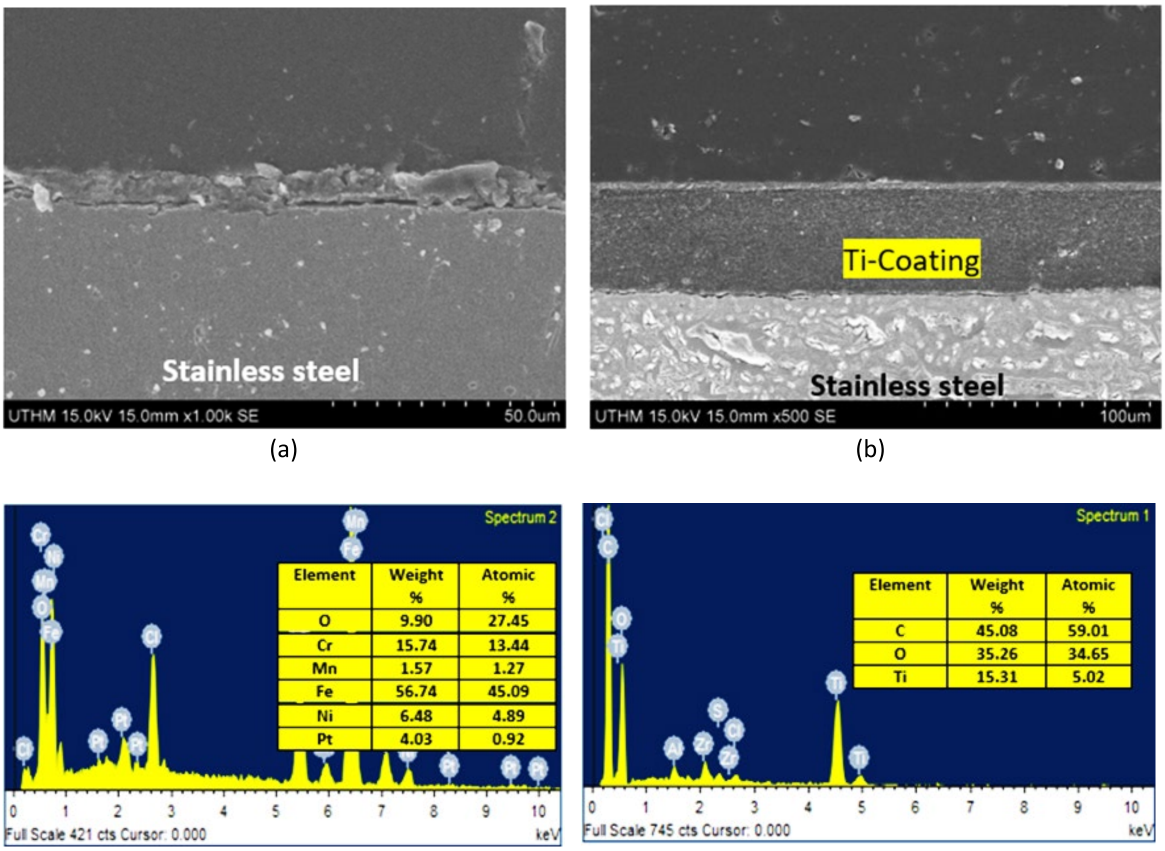


Fig. 3. The corrosion experiment setup

3. Results

The cross-section view of both untreated and treated 304 stainless steel (SS304) is shown in Figure 4. The average thickness of the titanium (Ti) coating applied during the phosphating process was 65.62µm using optical microscopy (OM). The existence of Ti coating element was analysed and confirmed by EDX spectrum analysis as shown in Figure 4(d), where the Ti element showed a reading of 15.31 wt%, while oxygen (O) showed a reading of 35.26 wt%. Meanwhile, the untreated sample also confirmed a 304 stainless steel due to the detected element, as illustrated in Figure 4(c). The Ti coating functions as a protective barrier, inhibiting direct contact between the Fe metal and the corrosive environment, thereby having the potential to decrease the corrosion rate. The observation has shown that Ti coatings may successfully prevent the corrosion of Fe-based metals by creating a durable and protective oxide layer on the surface, such as TiO_2 [18]. This statement was consistent with the corrosion analysis conducted for untreated and treated 304 stainless steel in the following section.



Element	Weight %	Atomic %
O	9.90	27.45
Cr	15.74	13.44
Mn	1.57	1.27
Fe	56.74	45.09
Ni	6.48	4.89
Pt	4.03	0.92

Element	Weight %	Atomic %
C	45.08	59.01
O	35.26	34.65
Ti	15.31	5.02

(a) (b)

(c) (d)

Fig. 4. SEM micrography of 304 stainless steel cross-sectional view (a) Untreated (b) Treated (c) EDX for stainless steel (d) EDX for a coating layer

Figure 5 illustrates the potentiodynamic polarisation curves of the samples following open-circuit experiments in a 6M KOH solution. Significant disparities in both the corrosion potential (E_{corr}) and corrosion densities (I_{corr}) may be observed between the untreated and treated samples listed in Table 3. The untreated sample has a greater corrosion current (Icorr) value of 1.59×10^{-5} A and a lower corrosion potential (E_{corr}) value of -0.63842 V, resulting in a corrosion rate of 0.007109 mm/year. In comparison, the treated sample has a lower Icorr value of 5.25×10^{-6} A, a higher E_{corr} value of -0.64753 V, and a corrosion rate of 0.002349 mm/year. A metal with a lower corrosion density, I_{corr}, and a higher corrosion potential, E_{corr}, indicates more excellent corrosion resistance. Higher values of the corrosion current density (I_{corr}) and lower values of the corrosion potential (E_{corr}) suggest reduced corrosion resistance in metals [19].

According to Ariza-Figueroa *et al.,* [20], stainless steel of AISI 304 with I_{corr} values below 0.1 μA/cm^2 has shown a significant resistance level against corrosion. Typically, metals with lower E_{corr} values and higher I_{corr} values have superior corrosion resistance [21-23]. Furthermore, higher E_{corr} and lower I_{corr} values indicate a decelerated corrosion process, which signifies enhanced corrosion resistance [24]. Introducing specific inhibitors can decrease E_{corr} and increase I_{corr} values, suppressing reactions on the material's surface and improving its corrosion resistance [25]. Furthermore, lower E_{corr} values and higher I_{corr} values in uncoated materials suggest a higher corrosion rate, underscoring the need for protective coatings [26].

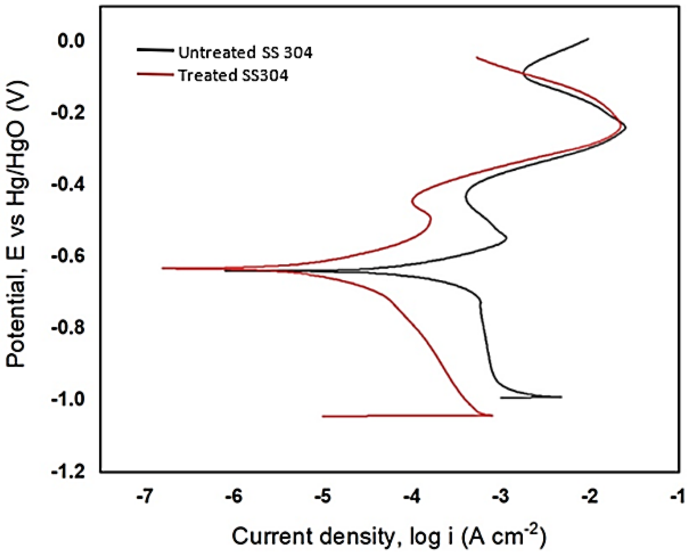


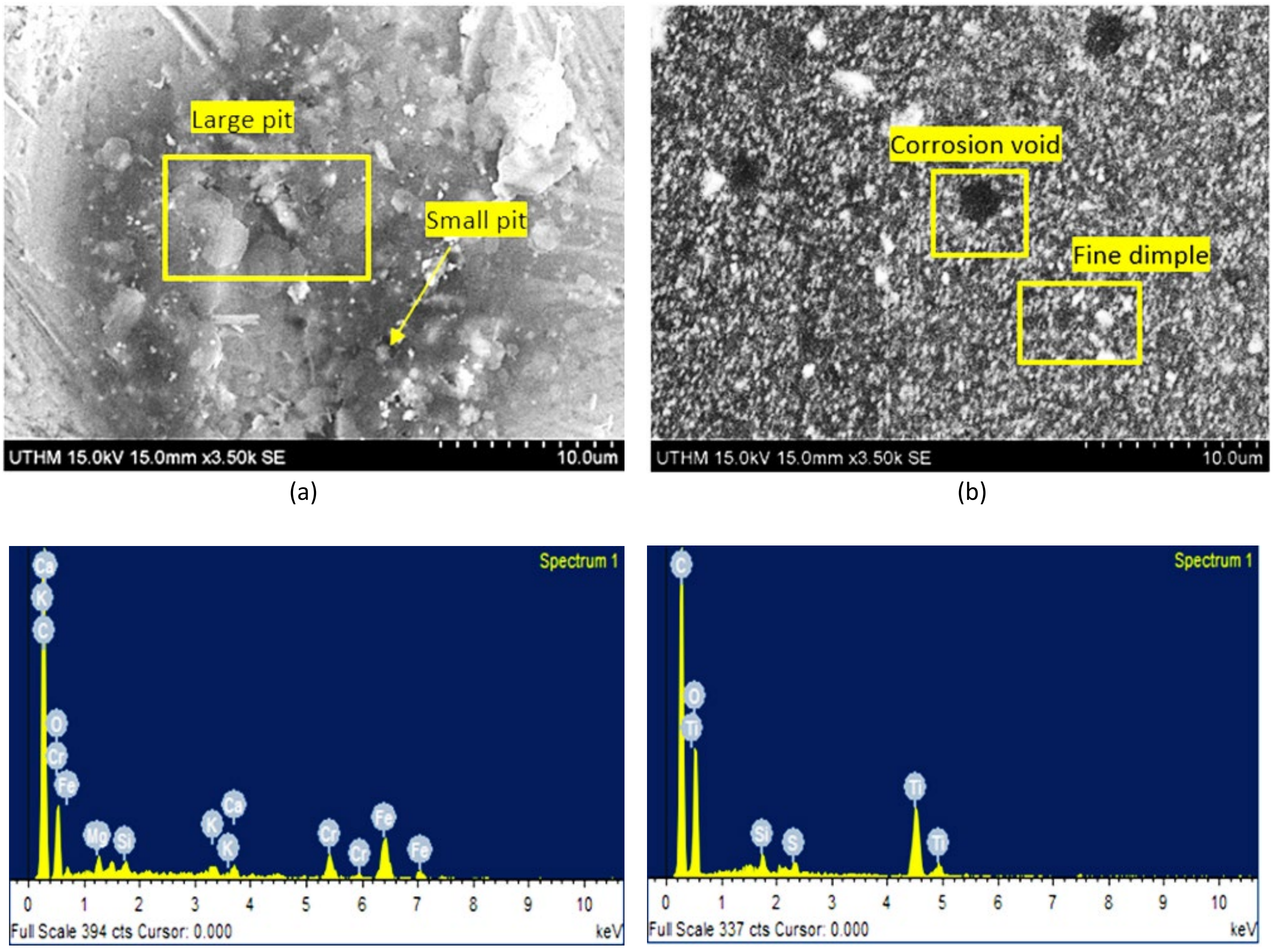
Fig. 5. Tafel plot for SS304

Table 3
The acquired corrosion analysis parameters

	Untreated SS304	Treated SS304
I_{corr} (A)	1.59×10^{-5}	5.25×10^{-6}
E_{corr} (V)	-0.63842	-0.64753
Corrosion rate (mm/year)	0.007109	0.002348

Essentially, the correlation between the rate at which corrosion occurs (corrosion current density, I_{corr}) and the tendency for corrosion to happen (corrosion potential, E_{corr}) in metals is of utmost importance in evaluating their ability to resist corrosion. Higher values of I_{corr} and lower values of E_{corr} generally indicate less corrosion resistance, whereas lower values of I_{corr} and higher values of E_{corr} imply an elevated vulnerability to corrosion. The permissible corrosion rate for iron-based metal in alkaline settings may vary depending on the application and environmental circumstances. According to the existing literature [27], pure iron has been observed to have a corrosion rate of around 0.25 ± 0.02 mm/year in Hanks' Balanced Salt Solution (HBSS). The corrosion rate mentioned is a benchmark for the allowable range of iron-based metals in alkaline solutions. Hence, it can be deemed appropriate for Fe-based metal to have a corrosion rate of around 0.25 mm/year in alkaline conditions, as indicated by studies that conducted immersion experiments in HBSS [27]. The treated sample demonstrated superior corrosion resistance with a corrosion rate of 0.002348 mm/year compared to the untreated sample with a 0.007109 mm/year corrosion rate. This extends the service life of SS304 treated samples by preventing contact with corrosive substances. The coating layer may be responsible for its exceptional corrosion resistance.

In addition, the microstructure of the corroded surfaces revealed deterioration of the smoothness of the sample's surface, which was examined through SEM and EDX analysis. This indicated the penetration of ions into the material surface, thus forming the corrosion products for untreated and treated SS304 as shown in Figure 6. According to Vasily *et al.,* [28], the pit morphology depends on alloy and environmental composition. The pit can take different forms, either hemispherical, dish-shaped, flat-walled, depending on crystalline structure, or even an arbitrary shape. Besides, the pit mouth can be open (uncovered), have a lacy metal cover, or a cover consisting of corrosion products. In the case of f 304 stainless steel in KOH solution, it is clearly seen that many pits were observed for untreated SS304 surface including a large pit mouth (uncovered). Contradicted with treated SS304, no pit was detected at the surface because Ti-coating gives the substrate extra protection. However, the potential pits to appear can be seen as fine dimples and corrosion voids, as represented in Figure 6(b). Thus, the metallic coating applied on the SS304 efficiently increases the corrosion resistance when exposed to alkaline conditions.



(a) (b)

(c) (d)

Fig. 6. SEM morphology on the corroded surface of 304 stainless steel (a) Untreated (b) Treated (c) EDX spectrum for pits area (d) EDX spectrum for the void area

4. Conclusions

The corrosion behaviour of untreated and treated 304 stainless steel in 6M KOH electrolyte has been investigated. The results demonstrated a considerable gain in corrosion resistivity for the treated samples compared to their untreated counterparts, with the treated sample demonstrating a particularly impressive corrosion rate of 0.002348 mm/year and 0.007109 mm/year, respectively. This improvement can be linked to Ti-based coating with a curing time of 10 minutes, as proven by the extensive investigation of microstructures and corrosion behaviours. Regarding surface microstructures, untreated samples showed small and large pits, while no pit was detected for treated samples. Only fine dimples and voids were observed for the treated sample. Even though untreated SS304 have pits, the corrosion rate value is still considered excellent and accepted for stainless steel because the corrosion rate penetration is below 1 mils per year (mpy).

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