

PTFE THIN FILM COATING ON 316L STAINLESS STEEL FOR CORROSION PROTECTION IN ACIDIC ENVIRONMENT

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ABSTRACT

Stainless steels (SS) are the most common materials used in various engineering applications because of their fine mechanical and excellent corrosion resistance properties in different mediums. 316L SS shows poor corrosion behavior in HCL. Hard coating is often required for protection of 316L SS from wear and corrosion. In this research paper polytetrafluoroethylene (PTFE) was coated on 316L SS by spin coating technique to modify their corrosion resistance in acidic media containing Hydrochloric acid (HCL). The anticorrosion property of the 316L SS and PTFE coating was studied in 40% HCL by electrochemical corrosion test and potentiodynamic polarization curves at room temperature and compared. The morphology of uncoated and coated substrates were examined by scanning electron microscopy (SEM) while the compositional analysis performed through energy dispersive x-ray spectroscopy (EDX). The morphology of the coated and uncoated substrates were also studied before and after electrochemical corrosion test and then compared. The thickness of the coating was also examined well. The result shows the remarkable improvement in the corrosion resistance of PTFE coating by decreasing the corrosion current density in HCL media.

KEYWORDS: *Polytetrafluoroethylene (PTFE), 316L Stainless Steel, Corrosion Resistance, Hydrochloric Acid (HCL)*

INTRODUCTION

Stainless steels (SS) are the most common materials used in various engineering applications due to their excellent mechanical and corrosion resistance properties in different environments and are also used as biomaterials production in various medical devices¹. The corrosion resistance of SS is provided by the formation of an oxide film with on the metallic surface containing Cr₂O₃ for 316L SS^{2,3}. However, under some environments the passive layer of SS may be lost and the failures are caused by pitting corrosion and due to the presence of chloride ions Cl⁻ it enhanced the metal corrosion through the passive layer⁴⁻⁷. 316L SS shows poor corrosion behavior and stability towards Hydrogen Chloride (HCL)⁸ therefore have active pitting corrosion behavior in HCL media. The corrosion of metals is an electrochemical process in an aqueous solutions. Corrosion mechanism of steel in HCL is previously discussed by M.N. Rahuma et al⁹ and the electrochemical reaction of iron corrosion in aqueous solutions containing Cl⁻ ions.

Different researchers worked on development of the resistive coating for corrosion prevention. G.X. Shen et al.¹⁰ prepared a smooth and uniform Titania (TiO₂) nanoparticles using sol-gel method to improve the corrosion resistance. D. Sidane et al.¹¹ introduced the thin

layer of TiO₂ on hydroxyapatite (HAP) sol-gel coatings. E. Marin et al¹² developed nanometric Al₂O₃ and TiO₂ alternating composite layers on 316L SS substrates via Atomic Layer Depositions (ALD) to decrease corrosion rate. D. Gopi et al¹³ coated Polypyrrole/strontium hydroxyapatite (PPy/Sr-HA) bilayer on SS by the electropolymerisation of pyrrole. P. Ju et al¹⁴ developed a double coating of Pd-Ni/Pd-Cu by electroplating. J. Mondal et al¹⁵ developed a nanometric aluminum and titanium oxides alternating laminates layer of ceramics and deposited by ALD process.

The above literature review reveals that previous studies were focused on developing a corrosion resistive coating in salt solution. The objective of this work is to develop a corrosion resistive coating which resist it in acidic environment.

Polytetrafluoroethylene (PTFE) is a representative of the class of polymers and has great importance due to its wide range applications which has attracted considerable interests characterized with high chemical inertness, good hydrophobic, antifriction properties, self-lubricating properties and high temperature resistance¹⁶⁻¹⁸. PTFE is chemically inert and has a relatively high melting temperature of 325°C and has excellent nonstick properties¹⁹.

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The aim of the present work is to deposit a thin film coating of PTFE on 316L SS and to investigate the corrosion behavior in HCL solution, moreover the study is further extended to compare the changes in the surface morphology of the coating before and after electrochemical corrosion test.

MATERIALS AND METHODS

Sample Preparations

The 316L SS samples were separated from the sheet having dimensions of 15 x 15 x 1 mm³. Then the samples were mechanically polished with 80-1000 grit SiC papers for better surface for coating and then rinsed with distilled water. Before coating the substrates were dipped in acetone and placed in ultra sonicator to remove residual grease and contaminations for 30 min, rinsed

with distilled water and then dried at room temperature.

Coating of substrate

A PTFE thin film was deposited on the 316L SS substrate through spin coating technique. In spin coating procedure small drop of fluid resin was deposited onto the center of the substrate that is spinning at a high speed (approx. 3000 rpm), centripetal acceleration will let it to scattered off to the boundaries of the substrate forming a thin film on the surface of the substrate. Substrate was attached to the spin coater with double stick tape and rotated around 3000 rpm and 5 drops of PTFE was deposit onto the center of the substrate leaving a uniform layer on it. To reduce the stresses and making it crystalline the samples were then annealed in a tube furnace at 250°C for two hours and relax the PTFE film onto SS.

Table 1. Elemental Composition of 316L Stainless Steel

Chemicals	C	Mg	P	S	Si	Cr	Ni	Mo	N	Fe
Compositions (%)	0.03	2	0.045	0.03	0.75	16-18	10-14	2-3	0.10	Bal



Fig 1. Spin coating step procedure

Characterizations

The surface morphology of the coating and uncoated substrates were studied through JSM5910 scanning electron microscope (SEM) while compositional analysis performed through energy dispersive x-ray spectroscopy (EDX) equipped with SEM. Moreover, the changed on the surface morphology of the uncoated and coated substrate were also studied after electrochemical corrosion test and well compared with the surface morphology before electrochemical corrosion test. This comparison gives us the changes occur on their surface morphology. The thickness of the coating was also examined through SEM.

Chemical Stability

The chemical compatibility and stability of the PTFE

coated samples were examined by the long term immersion in the HCL solutions of different concentration (20%, 40%, 60% & 80%) for 120 hours. One side of the substrates were coated while the other side remained uncoated to compare the compatibility and changes occur after long term immersion. Both the sides (coated & uncoated) of the substrate were exposed to HCL solution with different concentrations covering both the layers.

Electrochemical Corrosion test

Electrochemical and Potentiodynamic polarization test were performed through gamry apparatus (gamry framework) to evaluate the corrosion rate of uncoated 316LSS and PTFE coating on 316L SS in 40% HCL solution and compared to study the reduction in the corrosion rate of the coated substrate. The uncoated area of the

substrate was soldered with a conducting copper wire and inserted in the narrow tube. The sample was mounted with epoxy in such a way that it cannot be exposed to the HCL solution leaving an area which is exposed to solution. Electrochemical experiments were carried out at room temperature using three electrode corrosion cell setup with uncoated and PTFE coated sample of 1.43 cm² exposed area as working electrode, saturated calomel electrode (SCE) as reference and graphite as counter

electrode which are further connected with potentiostat. Polarization measurements were performed with a scan rate of 1mV/s at a potential initiated at - 400 mV to + 400 mV versus corrosion potential after 60 min exposure to the test electrolyte for achieving a stabilized potential. Corrosion potential (E_{corr}), corrosion current density (I_{corr}) and corrosion rate (C.R) were determined using the Tafel diagram and potentiodynamic polarization curves. Figure 2 shows the three electrode corrosion cell system.

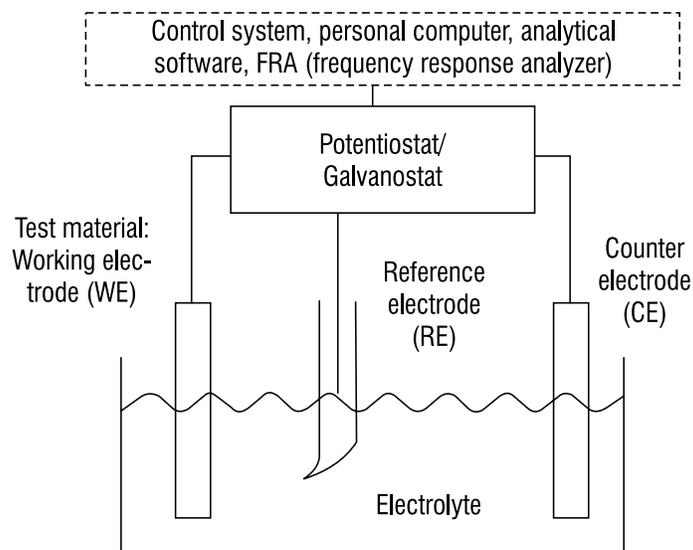


Fig 2. Three electrode corrosion cell system

RESULTS AND DISCUSSION

Microstructure

The morphology of the bare and coated substrate were studied through SEM at different magnification levels and are compared. Figure 3 shows the morphology of uncoated or 316L SS substrate, PTFE coated substrate, uncoated substrate after electrochemical corrosion test, PTFE coated substrate after electrochemical corrosion test respectively. The thickness of the coating measured was approximately 30 μ m. The PTFE coating shows uniform distributed layer onto SS.

The compositional and elemental analysis of the PTFE thin film coating on 316L SS was performed through EDX equipped SEM. Figure 4 and table 2 shows the peaks and elemental compositions of PTFE respectively.

Damage surface was observed on uncoated sample

after electrochemical test showing the corrosion layer over the surface of the substrate showing damaged and thick corrosive layer indicating unprotected surface. While in comparing with the coated substrates it shows some pits present on the surface hence more protected surface than uncoated substrate.

The compositional inspection of the PTFE thin film coating performed through EDX result shows the peaks of Carbon (C), Fluorine (F), Silicon (Si), Sulphur (S), Titanium (Ti) and Iron (Fe). EDX test confirms the presence of PTFE in the coating. Si, S and Ti can also be seen in small quantities in the PTFE coating. This may be present due to some impurities.

Chemical Stability of the Substrates

The chemical stability of the substrates were studied by immersing the substrate for long time up to 120hr in such a way that one side of the substrate is coated

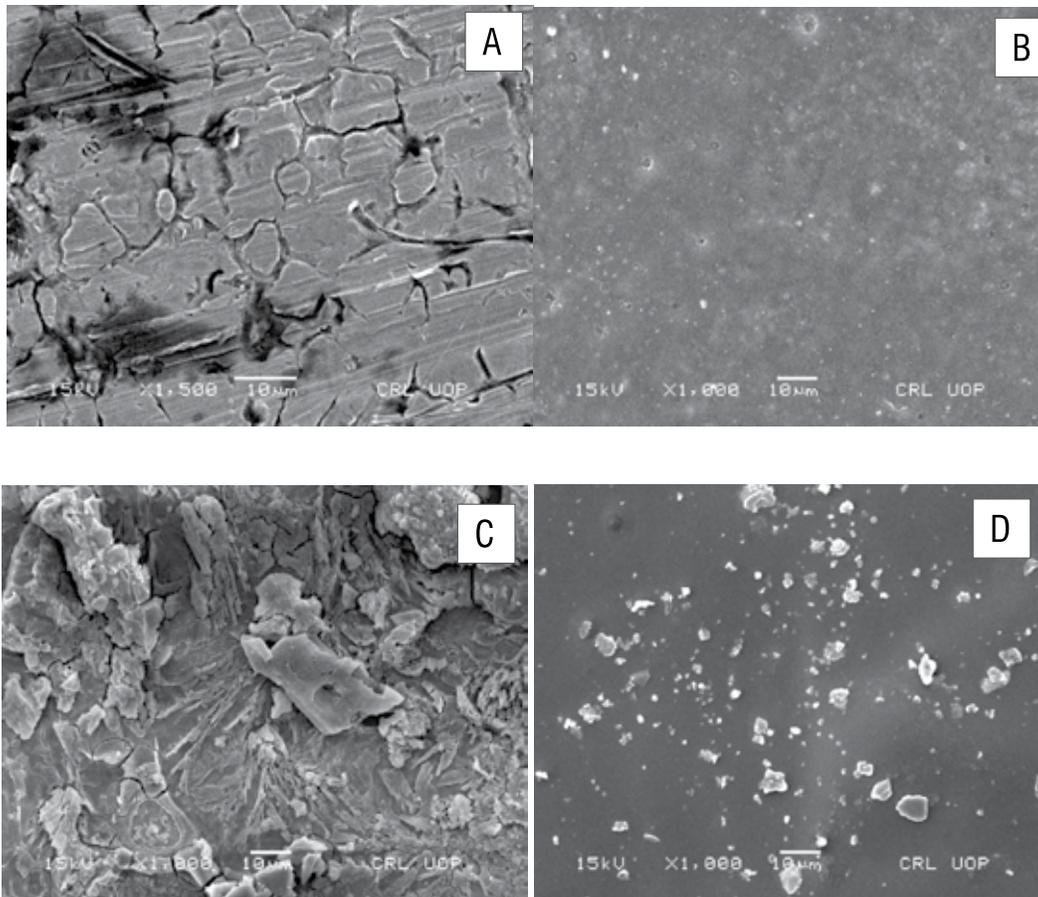


Fig 3. SEM Micrograph of (A) Uncoated substrate (B) PTFE coated substrate (C) Uncoated substrate after electrochemical corrosion test (D) PTFE coated substrate after electrochemical corrosion test

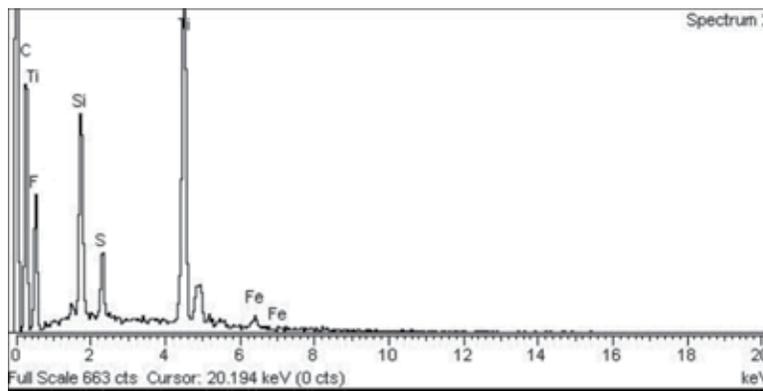


Fig 4 EDX peaks of PTFE coating

Table 2. Elemental compositions of PTFE

Elements	C	F	Si	S	Ti	Fe
Compositions (Wt. %)	59.83	7.11	18.64	2.98	9.65	1.79

while the other side is kept uncoated and immerse in the HCL solution of different concentrations of 20%, 40%, 60% and 80% covering both the layers of the substrate.

After long term immersion for 120hr the coated area was remained protected while the uncoated or bare area shows the active corrosion in HCL solution indicating pitting corrosion behaviour. 20% HCL solution shows

light corrosion and occurs after a long time, 40% and 60% shows more corrosion as compared to 20% HCL and corrosion occurs after 24 hours while 80% HCL solution shows harsh corrosion and started after few hours. A comparison between the coated and uncoated area of the substrates in various HCL concentrations is shown in Fig 5.

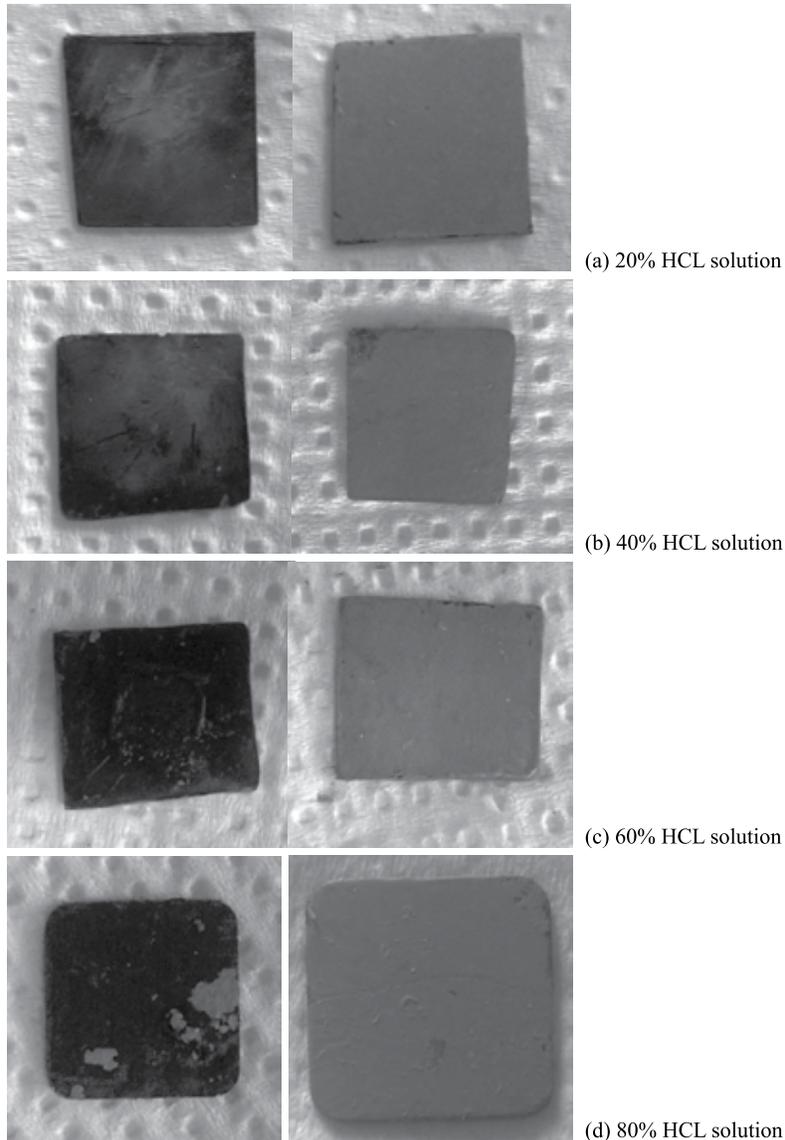


Fig 5. Chemical Stability of coated and uncoated substrates in various HCL concentrations

Corrosion Measurement

Electrochemical corrosion test was performed on uncoated 316LSS and PTFE coating in 40% HCL solution. The potentiodynamic polarization curves of uncoated 316L SS, PTFE coated on 316L SS and their comparison shows in figure 6, 7 and 8 respectively. The electrochemical data of 316L SS substrate and PTFE coated substrate from polarization test in 40% HCL

solution shown in table 2. From the results it can be seen that 316L SS exhibited a corrosion potential (E_{corr}) of -258.651 mV and corrosion rate (C.R) of 2.976449×10^{-5} mpy. However PTFE coating was found to be much nobler, corrosion potential (E_{corr}) of -379.651 mV and corrosion rate (C.R) of 4.013165×10^{-6} . Thus from this comparison of C.R we can conclude that PTFE coating decreases the corrosion rate by 86.5% when compared to bare 316L SS substrate in HCL solution.

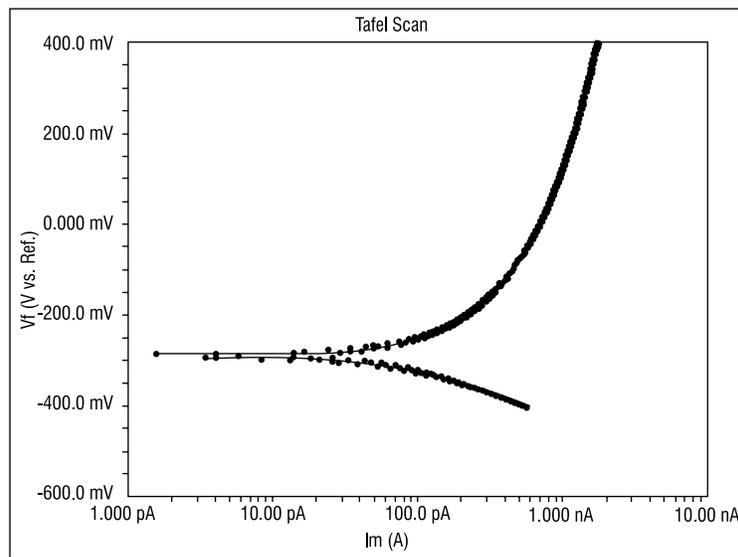


Fig 6. Potentiodynamic Polarization curve of 316L SS substrate in 40% HCL

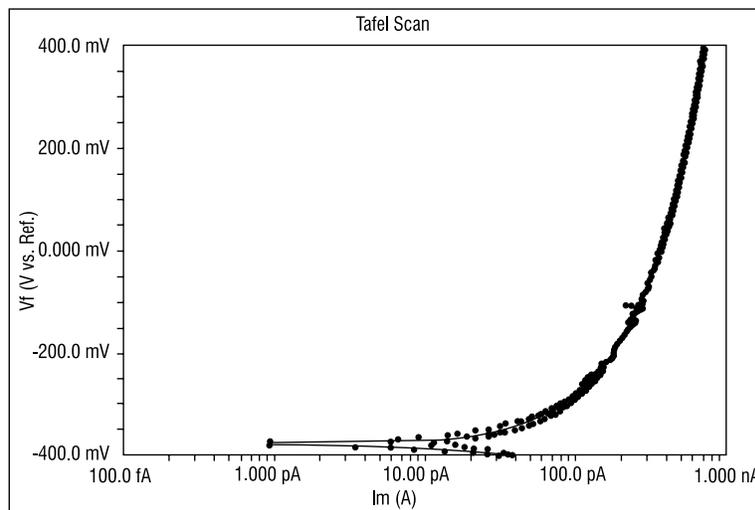


Fig 7. Potentiodynamic Polarization curve of PTFE coated substrate in 40% HCL

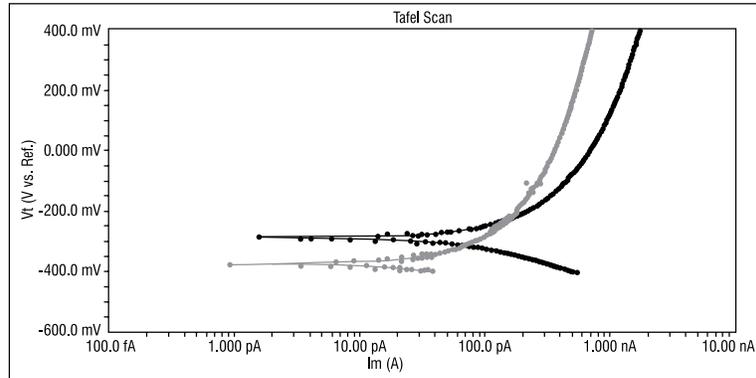


Fig 8. Comparison of Potentiodynamic Polarization curves of coated and uncoated substrates in 40% HCL

Table 3. Electrochemical data of 316L SS and PTFE coating

Specimen	E _{corr} (mV)	I _{corr} (pA)	C.R (mpy)
316L SS	-258.651	105.7	2.976449 X 10 ⁻⁵
PTFE coating	-379.651	14.25	4.013165 X 10 ⁻⁶

CONCLUSION

A uniform and smooth thin film PTFE coating on 316L SS was successfully deposited through spin coating technique. No cracks or discontinuities were observed by SEM analysis on the coating before and after electrochemical corrosion test but very small pores were detected on the sample. The electrochemical corrosion and potentiodynamic polarization test performed in 40% HCL at room temperature revealed that PTFE coating on SS was successful in providing superior corrosion resistance by decreasing the corrosion rate by 86.5% in HCL solution when compared with the bare sample. Thus PTFE coating shows great potential to be used in harsh acidic environment where high corrosion resistance is requested.

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