The corrosion resistance of 316L stainless steel coated with a silane hybrid nanocomposite coating

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Abstract

The present work aims at evaluating the corrosion resistance of 316L stainless steel pre-treated with an organic–inorganic silane hybrid coating. The latter was prepared via a sol-gel process using 3-glycidoxypropyl-trimethoxysilane as a precursor and bisphenol A as a cross-linking agent. The corrosion resistance of the pre-treated substrates was evaluated by neutral salt spray tests, linear sweep voltammetry and electrochemical impedance spectroscopy techniques during immersion in a 3.5 % NaCl solution. In addition, the effect of the drying method as an effective parameter on the microscopic features of the hybrid coatings was studied using Fourier transform infrared spectroscopy and scanning electron microscopy. Results show that the silane hybrid coatings provide a good coverage and an additional corrosion protection of the 316L substrate.

Keywords: 316L stainless steel; corrosion protection; drying methods; hybrid coating; silane

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1. Introduction

One of the most effective metal corrosion control techniques consists of the electrical isolation of the anode from the cathode [1-2]. A chromium oxide (Cr_2O_3) passivation layer formed on the surface of stainless steel in oxidizing environments is a typical example of this and guarantees the durability and the corrosion resistance of this particular metal [2-4]. However, chromates are now heavily restricted in corrosion control procedures due to the Cr(VI) toxicity and their carcinogenic nature [5]. For this reason, a number of promising candidates, so called “green inhibitors”, have been explored with the hope of replacing chromates. Among them, silanes, a group of silicon based organic-inorganic chemicals, have emerged as a very promising alternative [6].

Silane films not only ensure the adhesion between metal substrates and organic coatings but they also provide a thin, but efficient, barrier against oxygen diffusion to the metal interface [7-8]. Recently, silane coatings have attracted the attention of the nanotechnology industry because they provide a highly uniform, robust and reliable coating with lateral resolution on the nanometer scale [9]. A general silane structure is $(\text{XO})_3\text{Si}(\text{CH}_2)_n\text{Y}$, where XO is a hydrolysable alkoxy group, which can be methoxy (OCH_3), ethoxy (OC_2H_5) or acetoxy (OCOCH_3). Y is an organofunctional group such as epoxy, vinyl ($\text{C}=\text{C}$) or amino (NH_2) which is responsible for a good adhesion of a silane treated metal surface [6, 10].

3-glycidoxypropyl-trimethoxysilane (GPTMS) is one of the organofunctional silane molecules that have been used as an effective coupling agent or adhesion promoter in glass/mineral-reinforced polymeric composites for decades [6, 11–13]. Also it has been used in transparent abrasion-resistant hybrid coatings for polymers [14–17], metals [18–20] and for gas separation membranes [21, 22].

GPTMS can undergo a variety of reactions during the preparation of a hybrid by a sol-gel route. Hydrolysis of the methoxy groups gives silanol groups, which can subsequently

condense to form the silane network. The silicon atom in GPTMS is tri-functional in terms of the reactive methoxy groups and is therefore able to form a three-dimensional branched siloxane silane network with a nominal stoichiometry $\text{SiO}_{1.5}$. The epoxy rings can be opened and polymerised to form a linear poly(ethylene oxide) organic network [22, 23].

Cross-links between the two networks arise either from the pre-existing link in the GPTMS molecule, by direct reaction of silanols with epoxy rings, or by condensation of silanols with hydroxyl of the opened epoxy rings. The uncatalysed ring opening reaction occurs at a useful rate only at elevated temperature and so thermal curing is required [22, 23].

The use of the sol-gel process to prepare highly intermingled inorganic-organic hybrid polymer networks is of current scientific interest since it offers the possibility of tailoring the material properties by variation of the relative composition of the inorganic and organic phases. With such systems, the inorganic and organic networks are formed together (often simultaneously) to achieve homogeneous phase morphologies, which are impossible to produce by the other routes [22].

The microscopic features of the hybrid coatings obtained with sol-gel process depend on those hydrolysis and condensation reactions that are generally controlled by the pH of the solution [24-25]. In the acid catalyzed reaction, the hydrolysis step is faster than the condensation step, resulting in a more extended and less branched network structure. In the base catalyzed reaction, condensation is faster than hydrolysis, resulting in highly condensed species that may agglomerate into fine particles [24, 26-28]. Furthermore, the sol-gel process includes a very complex reaction involving many variables such as pH, type and amount of solvent, water/alkoxide ratio [28, 29], concentrations of organic [26] and inorganic [23] reactants, aging [30], cross-linking agent/alkoxide ratio, and drying methods. We investigated some of these factors in previous studies on a 1050 aluminium alloy [23, 26, 28-29, 31].

As a follow-up of these results, the present work aims at evaluating the structure and corrosion protection performance of silane sol–gel hybrid coatings deposited on austenitic 316L stainless steel. This steel combines properties such as acceptable biocompatibility and excellent mechanical resistance with easy fabrication at low cost, which permits its use in many industrial applications, consumer products and temporary orthopedic devices [32-33]. In general 316L stainless steel presents good anti-corrosion properties, however its corrosion resistance is weakened when it is subjected to a medium containing chloride or moisture environment [34-35], with a tendency to suffer localized corrosion (e.g. pitting corrosion). The corrosion may result in the loss of aesthetic appearance and structural integrity [36], and can be accompanied by the release of potentially toxic ions, which can be reduced by protective coatings [37-39]. Furthermore, the corrosion of stainless steel welds and joints occurs in the transpassive potential region in highly oxidizing environments of many industrial processes, especially in sulfuric acid media releasing Cr(VI) ions [40]. To resolve all these problems, the application of hybrid coatings has been increasingly investigated in the last years [38,41–43].

Sol–gel coatings were prepared from hydrolysis and condensation of 3-glycidoxypentyltrimethoxysilane (GPTMS) as the precursor and bisphenol A (BPA) as the cross-linking agent in acid catalysed condition. The effect of the drying method as an effective parameter on the microscopic features and morphology of the silane hybrid coating was examined using Fourier transform infrared spectroscopy (FTIR). In order to study the corrosion protection efficiency, the coated and uncoated steel samples were investigated using potentiodynamic polarization curves, neutral salt spray tests and electrochemical impedance spectroscopy. In addition, scanning electron microscopy (SEM) analysis was performed to visualize the surface.

The results show that pre-treatments based on silane hybrid solutions present improved corrosion resistance for the 316L substrates. Furthermore, the methodology proposed in this work is simple to apply and it is compatible with actual environmental concerns.

2. Experimental

2.1. Materials

All chemicals and reagents used were purchased from Merck, including 3-glycidoxypentyltrimethoxysilane (GPTMS), bisphenol A (BPA), hydrochloric acid, sodium chloride and 1-methylimidazole (MI).

2.2. Preparation of the sol

The sol was prepared by adding stoichiometric amounts of the silane precursor (GPTMS) and the organic cross-linking agent (BPA) into HCl-acidified water ($\text{pH} = 2$). The $\text{H}_2\text{O}/\text{Si}$ molar ratio was chosen to be 0.5, relying on results of a previous study [26]. The solution was stirred at ambient temperature for 80 minutes at a rate of 240 rpm. Then MI was added to accelerate the condensation reaction after hydrolysis. The MI/Si molar ratio was 0.01. The solution was stirred again for 10 minutes at ambient temperature. The result was a clear and colourless homogenous solution.

2.3. Substrate preparation and coating deposition

The substrate material used for the present investigation was 316L stainless steel alloy, purchased from Watrin-sprl CO. Plates ($7\text{ cm} \times 15\text{ cm} \times 0.1\text{ cm}$) were used for the salt spray tests, while coupons (4.98 cm^2 area and 0.1 cm thickness) were used for all other experiments. The substrates were initially degreased with detergent and then polished with 600 and 1200 grit sand papers. The polished samples were rinsed with distilled water and degreased with 1 M NaOH solution at $50\text{ }^\circ\text{C}$. Consequently the substrates were neutralized with 0.01 M HCl

solution to remove excess of NaOH solution. As a last step, the substrates were cleaned with acetone to remove all dirt and grease and were then air-dried.

The cleaned substrates were dipped into the sol for a duration of 5–10 s. In order to study the effect of drying method on the microscopic features and morphology of the hybrid coatings, a series of the silane-treated substrates was dried at the ambient temperature for 2 weeks, denoted as air dried silica hybrid coating (A-SHC), while another series was dried at ambient temperature for 24 hours and placed in a furnace to cure at temperatures ranging from ambient (25 °C) to 130 °C for 90 minutes to extensively crosslink the silane films. This series of the coatings is denoted as thermal cured silica hybrid coating (T-SHC).

2.4. Characterization techniques

2.4.1. FTIR measurements

FTIR measurements were carried out using a Bio-Rad 575C spectrophotometer in the mid-IR range from 4000 to 400 cm^{-1} . All spectra were obtained at an incident angle of 45 degrees normal to the surface of the specimen, with a spectral resolution of 4 cm^{-1} . For each measurement 64 scans were collected.

2.4.2. Scanning electron microscopy

Scanning electron microscopy (SEM) measurements were performed on the surface of coated substrates to characterize the surface morphology with a XL30 SEM microscope. Secondary and backscattered electron images were collected at 15 kV.

2.5. Corrosion tests

2.5.1. Anodic and cathodic polarization tests

Anodic and cathodic polarization tests were carried out on 316L stainless steel substrates with and without the silane treatments at 25 °C in a neutral 3.5 % NaCl solution. Prior to the

measurements, in order to reach a steady potential, the silane treated samples were kept in the working solution for at least 3 hours. An Ag/AgCl electrode and a platinum mesh were used as the reference and counter electrodes, respectively. The potentiodynamic measurements were taken within the range of -1500 to 1000 mV versus Ag/AgCl at a rate of 1 mV/s.

2.5.2. Salt spray testing

The corrosion resistance of the coated substrates was evaluated in a neutral salt spray test, following the ASTM B117 standard and employing a 5 % sodium chloride solution. Prior to exposure, the backs and edges of the specimens were covered with adhesive tape. An artificial scratch was made in the coating that penetrated to the alloy substrate to examine the delaminating behavior. The specimens were removed from the salt spray chamber after weekly intervals and representative regions were imaged with a digital camera. The images were used to evaluate the corrosion performance by determining the time of exposure to the first occurrence of corrosion, to measure corrosion creep from the artificial scratch and to evaluate coverage with corrosion products.

2.5.3. Electrochemical impedance spectroscopy measurements

Electrochemical impedance spectroscopy measurements (EIS) were employed to monitor the corrosion performance of the silane treated 316L stainless steel substrates in a 3.5 % NaCl solution. EIS measurements were carried out at the open circuit potential, using an Autolab PGStat 20 instrument. The data were obtained as a function of frequency, using a sine wave of 10 mV amplitude peak to peak. A frequency range of 10^5 Hz to 10^{-2} Hz was selected. A three-electrode electrochemical cell arrangement was used, consisting of the working electrode (4.98 cm² of exposed area), Ag/AgCl electrode as reference and platinum as counter electrode.

3. Results and discussion

3.1. Characterization

Figure 1 shows the FTIR spectra of two series of hybrid films (A-SHC and T-SHC). The most resolved bands are attributed to the vibration frequencies of the glycidoxypopylsiloxane structural fragment. They include the oxiranes methylene bending at 1480 cm^{-1} , the epoxide ring breathing band at 1250 cm^{-1} and the antisymmetric epoxide ring deformation bands at 758 and 902 cm^{-1} [44]. The band of the antisymmetric epoxide ring deformation at 758 and 902 cm^{-1} in the magnified region from 729 to 950 cm^{-1} in panel A, appears to be intense. It allows the monitoring of the chemical reaction of the epoxy functional groups of organosilica-networks coupled with diol crosslinker groups. FTIR spectra of the cured film show the disappearance of this band, which indicates the formation of a cross-linked network under curing. Also the bands centered at 1100 cm^{-1} due to Si-O-Si [45] in the magnified region from 950 to 1200 cm^{-1} in panel B, are intensive as compared to that without curing. All these changes indicate the formation of a denser siloxane network upon thermal curing.

{INSERT Figure 1 HERE}

SEM was used to investigate the effect of the drying method on the morphology of the silica hybrid coatings. Figure 2 shows typical SEM micrographs of the T-SHC and A-SHC coatings. Cracking during drying is a rare event but could occur when the gel has had insufficient ageing and strength. After drying, the coatings were densified by heat treatment. During this step the residual stress grows until the point of generating some microcracks into the coating. Residual stress at the interface comes from the differential thermal expansion of both substrate and film coating. However, as soon as the corrosive medium penetrates through the layer and reaches the substrate, an electrochemical reaction takes place, causing an

increase of current density [46]. Fortunately, the evaluated coatings did not show such behavior and the surface appearance obtained by SEM characterization did not present any micropores and cracks in each of the samples. It seems that both of the coatings are uniform, defect and crack free.

{INSERT Figure 2 HERE}

3.2 Corrosion protection performance of the hybrid coating

The corrosion protection performance of the hybrid coatings was studied by linear sweep voltammetry (LSV). Figure 3 presents the potentiodynamic polarization curves of the bare and A-SHC and T-SHC coated 316L stainless steel substrates recorded after 3 hours of immersion in a neutral 3.5 % NaCl solution. Table 1 summarizes the electrochemical parameters obtained from these measurements. In comparison to the bare sample, the T-SHC coated sample shows a decrease in the corrosion current density, i_{corr} , around $10^{-8} \text{ A.cm}^{-2}$. In addition its potential is shifted to a higher value (-0.424 V). This coated sample shows an anodic behavior with limited current density in the range of $+0.55 \text{ V}$ over its E_{corr} , before the potential exceeds the breakdown potential. This behavior was improved by the protective barrier characteristic of the coating, as reported before [23]. However, the A-SHC coated sample into this solution did not significantly improve the corrosion resistance, but it showed some characteristics in the anodic current zone. In this way, the A-SHC coated sample presents a range of anodic potential at $+0.4 \text{ V}$ over the E_{corr} , before the point of the breakdown potential was reached. As a comparative result, the uncoated sample presented a continuous potential range characterized by a control mechanism of equilibrium in the oxidation and reduction reactions. Also a wide passive plateau in the potential characteristic of stainless steel was observed.

{INSERT Figure 3 HERE}

{INSERT Table 1 HERE}

Figure 4 shows the scanning electron micrographs for the bare and coated 316L stainless steel samples after potentiodynamic polarization in a neutral 3.5% NaCl solution. After the anodic polarization, a localized corrosion is observed for all samples, consisting of small cracks (Figure 4a and c) and pits (Figure 4e) of different sizes. A closer inspection shows that the region surrounding some of the cracks and pits is damaged (see Figures 4b, d and f). This may indicate a preferential localized attack, occurring after the electric potential exceeded the breakdown potential. This localized attack has resulted in debonding, delaminating and lifting of the coating from the substrate, possibly due to the hydrolysis reactions at the interface. Additionally, the diffusion of the oxidant ions speeds up and the corrosion rate increases causing an accumulation of the corrosion products at the interface, promoting the formation of bulges and even leading to microcracks as can be seen in Figures 4a and d. As the quantity of corrosion products increases, the protective coating is lost by the fact that the film peels off. Fractures of the coatings derived by the increasing of sweep over-potential initially grow from small cracking points or pitting in the coating. On the other hand, increasing towards higher potential promotes the cracking and peeling of the coating [46]. However, the coated samples show a higher corrosion resistance described by the E_{corr} and i_{corr} values (Table 1), as reported before.

{INSERT Figure 4 HERE}

The traditional salt spray coating corrosion resistance evaluation technique was used to investigate the corrosion performance of the sol–gel films. Pictures of the T-SHC and A-SHC

coated 316L stainless steel plates after 600 h of salt spray exposure are shown in Figure 5, along with bare plates as control (taped areas were cropped from the picture). It can be clearly seen that silane-treated surfaces do not exhibit any sign of corrosion and still retain their originally shiny surfaces. The bare panel, in contrast, exhibits early signs of corrosion. Therefore, these data confirm the good corrosion protection for a long term, of the silane sol–gel coatings.

{INSERT Figure 5 HERE}

Figure 6 presents the impedance spectra obtained for the bare and thermal cured hybrid coated 316L substrates (T-SHC) during immersion in a 3.5 % NaCl solution for a period of 43 days. For the bare sample, the EIS spectrum obtained after 5 hours of immersion is characterized by two overlapping time constants at a frequency around 10 rad.s^{-1} and another one at lower frequencies. The time constant at higher frequencies can be assigned to the presence of an oxide/hydroxide film existing on the bare 316L substrate, whereas the time constant at lower frequencies can be assigned to corrosion activity. The total impedance of the bare substrate after 5 hours of immersion is lower than $10^5 \Omega \text{ cm}^2$. At this stage, the bare surface shows a significant quantity of corrosion products.

For the coated substrate, the spectra are also characterized by the presence of two time constants. The high frequency time constant appears at 10^4 rad.s^{-1} and the low frequency time constant develops around $10^{-1} \text{ rad.s}^{-1}$. The results show that the hybrid coating increases the low frequency impedance of the system by about two orders of magnitude, in comparison to the bare sample. Moreover, for the coated sample, the total impedance values starts to decrease slightly as a function of immersion time and reaches a value of $6.727 \Omega \text{ cm}^2$ at the end of 37 days of immersion. The first signs of corrosion activity were detected between 37th

and 43th days of immersion. Nevertheless, the impedance values are more than 1.5 order of magnitude higher than those for the bare sample. These results indicate the protective nature of silane hybrid coating.

{INSERT Figure 6 HERE}

4. Conclusions

In this study, the corrosion protective behaviour of hybrid coatings based on 3-glycidoxypopyl-trimethoxysilane (GPTMS) as the precursor and bisphenol A (BPA) as the cross-linking agent as an “environmentally friendly” corrosion resistant protection system on 316L stainless steel substrates were studied. Additionally, the effect of drying method on microscopic features and morphology of the hybrid coatings was evaluated. The results of the FTIR analysis indicated the formation of a denser siloxane network upon thermal curing. In addition the SEM analyses confirm the formation of a transparent and homogenous film without any defect and micro crack especially cured at 130 °C. The results of the electrochemical analyses as well as salt spray test highlight the good barrier properties of the silane film. The pretreatment of 316L stainless steel with silane hybrid coating leads to the formation of a surface film that performs as a physical barrier on metal substrate. The presence of the silane film increases the anodic polarization of the system and its total impedance relative to the bare substrate. These results indicate the protective nature of silane hybrid coating.

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Table 1. Summary of the electrochemical parameters (corrosion potential, corrosion current, cathodic and anodic slopes) obtained from the polarization measurements in a 3.5 % NaCl solution.

Sample	E_{corr} (V)	I_{corr} (A.cm ⁻²)	B_c (V/dec)	B_a (V/dec)
Bare 316L	-0.564	1.35×10^{-6}	0.204	0.142
A-SHC	-0.423	2.84×10^{-7}	0.152	0.111
T-SHC	-0.424	7.63×10^{-8}	0.175	0.091

Figure Captions

- Figure 1. FTIR spectra of the silane films prepared under two drying condition: air exposed dried film (A-SHC) and thermal cured film (T-SHC); (A) magnified region from 729 to 950 cm^{-1} and (B) magnified region from 950 to 1200 cm^{-1} .
- Figure 2. Scanning electron images of the coated 316L stainless steel samples prior to polarization test: (a) T-SHC and (b) A-SHC coated 316L.
- Figure 3. Potentiodynamic polarization curves of the A-SHC and T-SHC coated and bare 316L stainless steel in a 3.5 % NaCl solution.
- Figure 4. Scanning electron images of the bare and coated 316L stainless steel samples after potentiodynamic polarization test in a neutral 3.5% NaCl solution with different magnifications: (a) and (b) T-SHC, (c) and (d) A-SHC and (e) and (f) bare 316L.
- Figure 5. A-SHC (a) and T-SHC (b) coated and bare (c) 316L stainless steel samples after 600 hours of exposure in the salt spray chamber.
- Figure 6. EIS Bode plots obtained for the bare and T-SHC coated 316L stainless steel substrates after different immersion times in a 3.5% NaCl solution.

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