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Effect of layer thickness on anticorrosive performance of 316L steel coated with bismuth-titanium films

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Abstract. Bismuth and titanium are elements with remarkable properties and applications in technological developments and in the field of biomedical engineering. The sol-gel method was used to form a bismuth-titanium system, which allowed to establish if it was possible to obtain films with anticorrosive properties on 316L stainless steel. The anticorrosive response was evaluated by means of Tafel curves, defining the parameters to obtain thin and functional films with good tribological properties. The coatings were obtained by the spin coating technique, varying the spin speeds from 3000 rpm to 5000 rpm with monolayer and bilayer systems. More positive values of corrosion potential were obtained when the steel is coated by the films, which implies a lower propensity to corrosion in saline medium, lower corrosion rates and higher potentials are reported for films with higher titanium content, likewise, better efficiency of the films with respect to the uncoated substrate is presented, the higher the titanium content and a strong influence between the calculated efficiencies and the centrifugation speeds is not observed.

1. Introduction

It is well known that coatings are applied to the surfaces of materials to help protect the material from the environment that produces corrosion or degradation reactions, improve appearance or provide insulation [1]. Corrosion is a defect that causes a deterioration in the properties of materials [2], especially metals, shortening the life cycle of use and causing a loss of efficiency in the applications for which it is estimated to be used, which is a technological and economic problem [3]. Coatings are used to insulate anodic and cathodic regions, they also prevent the diffusion of oxygen or water vapour that initiates corrosion or oxidation [4]. The characteristics of coatings are delimited by the use to which the material to be protected will be subjected.

Among some of the coating deposition techniques for the improvement of anti-corrosion properties are: electrodeposition, electrophoresis, sedimentation, thermophoresis, dip coating, spin coating, which are the most important ones [5]. In the present study, the spin-coating or centrifugation technique will be used. Likewise, the sol gel method has demonstrated a high efficiency in the development of thin films; this method consists of hydrolysis and condensation, originated from alkoxide precursors (organometallic compound), being this technique an alternative for the application of protective and thin films, with an efficient adhesion [6].

Therefore, the research question related to how to reduce the effects of corrosion in materials such as 316L steel was raised, and also the hypothesis of whether the variation of the rates in the production of coatings will affect the anticorrosive response of the material. Thus, the objective of this project was to contribute to propose an option to counteract the corrosion problem through the synthesis of a



bismuth-titanium (Bi-Ti) ceramic material by the sol-gel method, consolidating an anticorrosive protective film that would be effective on 316L stainless steel, evaluating its corrosion resistance properties; it is also a contribution to the generation of new academic knowledge on the quality of corrosion protection that Bi-Ti films can offer on 316L stainless steel substrates.

2. Methodology and materials

The methodological development of the project was based on three phases: the establishment of the chemical components and their concentrations for the preparation of the coatings, the film production or synthesis process and the centrifugation of the substances on the steel substrates.

The precursors used for this study were titanium (IV) butoxide - $\text{Ti}(\text{OBU})_4$ and bismuth (III) nitrate pentahydrate - $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$; as solvents, 2-ethoxyethanol and glacial acetic acid, $\text{C}_2\text{H}_4\text{O}_2$ were used and as complexing agent ethanolamine. Stainless steel 316 L (SS) parts with dimensions of $2\text{ cm} \times 2\text{ cm} \times 0.4\text{ cm}$ were used as substrates, which were polished with 360, 500, 1000 and 1200 silicon carbide abrasive papers and alumina. After the polishing phase and to remove impurities, the substrates were washed in acetone and in an ultrasonic bath for 10 minutes.

For the deposition of the films, the Bi-Ti solution was prepared by mixing bismuth nitrate and titanium butoxide in the presence of acetic acid. The synthesis of the sols was set up in the following sequence: first, 98% pure $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ was dissolved in CH_3COOH acetic acid (25 ml) under constant stirring for 2 hours at room temperature; then, 97% titanium tetrabutoxide (TBT) was added at different molar concentrations. Following preparation guidelines set out in the literature [7].

Figure 1 illustrates the experimental process developed to prepare and form the stable sols. In a first step, the components were mixed under magnetic stirring at room temperature and, in a second step, the substance obtained was filtered, obtaining the ideal viscosity for the stabilization of the solution. To deposit the coatings, a centrifugation mechanism was used on the samples, whose nomenclature can be seen in Table 1.

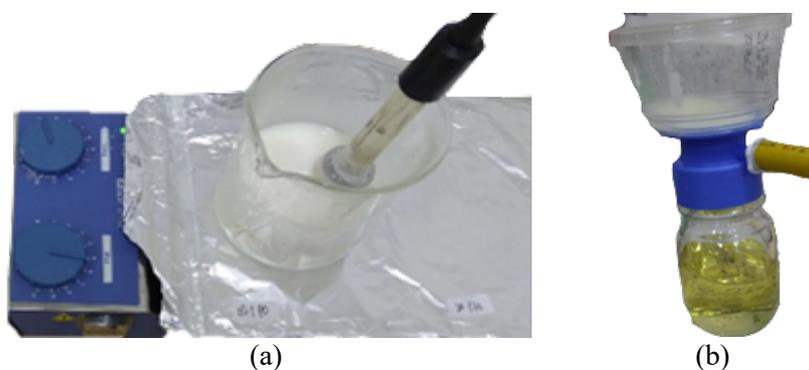


Figure 1. Stable solution preparation process; (a) magnetic stirring of the precursors in pH control; (b) filtration of the sol for the coatings.

Table 1. Characteristics of the samples used.

Sample	Spin speed (rpm)	Number of layers applied
S1	3000	1 and 2
S2	4000	1 and 2
S3	5000	1 and 2

3. Results and discussion

To evaluate the corrosion resistance of the specimens that were coated with two types of layer thickness (monolayer and bilayer) and varying the deposition rates, it was necessary to resort to the analysis of tafel curves, allowing to compare the progression of the corrosion rate and current of each of the configurations described above. The tafel curves correspond to a type of technique based on

physics concepts such as electric current, electrochemical reactions, polarization potentials, among others; being a key tool for approaching the subject of study.

Tafel curve analysis involves the determination of the slopes of β_a y β_c , which help to determine the active and passive characteristics of the corrosion system, such as corrosion potential, corrosion current and corrosion rate [8]. The plots in Figure 2(a), Figure 2(b), and Figure 2(c) reveal the Tafel polarization diagrams of the Bi-Ti system for the coatings at the speeds: 3000 rpm (S1), 4000 rpm (S2) and 5000 rpm (S3) respectively. The variation of the corrosion potentials of the films with respect to the corrosion potential of the substrate is observed.

The corrosion potential values tend to take positive values as the layer numbers, and thus the layer thickness, increase. When the corrosion potential values in coatings are more positive, there is less tendency to be affected by corrosion. In the particular case of the samples whose coatings were deposited at a speed of 3000 pm, as shown in Figure 2(a), a lower progression of the corrosion current is observed in the two-layer coating, which represents a better anticorrosion response in the 316L steel. Lower speeds imply higher coating thicknesses and thus better substrate protection, as was also demonstrated in [9].

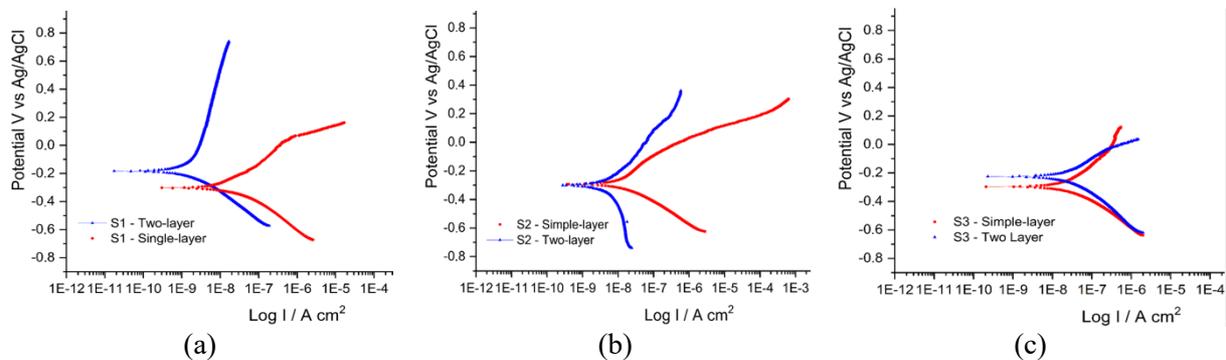


Figure 2. Tafel curve diagram for coatings centrifuged at (a) 3000 rpm, (b) 4000 rpm, (c) 5000 rpm.

In Figure 2(b) and Figure 2(c) respectively, the behavior of the difference between simple-layer and two-layer coatings is not as visually significant as shown in Figure 1; however, it can be seen that a greater layer thickness allows a decrease in the corrosion rate of the material; a situation that confirms the trend that has been shown in terms of improvement in the anticorrosion response. This behaviour is in line with studies that have demonstrated efficient corrosion protection mechanisms with coatings [10-12].

Table 2 shows the values of the anodic and cathodic slopes, corrosion currents and potentials of the coatings deposited on the steel samples used. Regarding the corrosion rate, it could be observed that the coatings represent protection mechanisms of the material, since the corrosion rate is decreased in comparison with the uncoated substrate.

Table 2. Current and corrosion potential data.

Variable	S1 Single layer	S1 Two layer	S2 Single layer	S2 Two layer	S3 Single layer	S3 Two layer	316 L Substrate
β anodic (V/ decade)	0.25393	1.05407	0.14991	0.21840	0.17483	0.24320	0.09947
β cathodic (V/ decade)	-0.21061	-0.20978	-0.16156	-0.15510	-0.18762	-0.18870	-0.07055
Corrosion potential (mV)	-0.293489	-0.175431	-0.193716	-0.290000	-0.222134	-0.293700	-0.186357
Corrosion current (nA)	2.599E-8	1.8408E-9	4.9783E-9	1.2415E-8	2.098E-8	2.540E-8	3.271E-7

4. Conclusions

The results of the corrosion evaluation using tafel curves show that all the coatings produced had better corrosion response conditions compared to the 316L steel substrate. The spinning speed significantly influences the corrosion properties. Generally, lower speeds favor higher film thicknesses and this allows better protection of the material. In relation to the number of layers, it could be shown that with two-layer coatings the corrosion rate is considerably reduced, especially at low rotational speeds.

According to the obtained results, it can be concluded that the corrosion potentials recorded for the films, without discriminating concentration and spinning speed, are more positive compared to the corrosion potential of the 316L substrate. It was possible to conclude that the tafel curves are a technique based on one of the branches of physics that allows obtaining data that can predict the corrosion rate of materials; being very important for its effectiveness the adequate application of the mathematical procedure of the identification of the anodic and cathodic regions.

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