



The influence of organophosphonic acid and conducting polymer on the adhesion and protection of epoxy coating on aluminium alloy



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ABSTRACT

In the present study we compared the beneficial effect on the incorporation of modified silane nanocoating and conducting polymer additive to the adhesion and protection of aluminium surface with epoxy coating. Results proved that the protection imparted by the silane coating, suitably modified with phosphonic groups, showed an excellent adhesion to the metallic substrate as well as good adherence with the epoxy outer layer, which result in an improved corrosion resistance. The incorporation of 1% of poly(3-methyl thiophene acetate) (P3TMA) also improved the adhesion force of the epoxy paint adhered to the aluminium surface. However, the adhesion force decreased with the increase of the immersion time in NaCl 3.5% solution. In conclusion, both bilayer systems [vinyltrimethoxysilane (VTMS)/tetraethylorthosilicate (TEOS) and VTMS/TEOS/ethylenediaminetetra(methylene phosphonic acid) (EDTPO)] and also the employment of P3TMA in the epoxy coating showed better results in accelerated corrosion assays, *i.e.* very low blistering formation was observed, compared with control samples based in a monolayer coating directly adhered to the aluminium surface.

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1. Introduction

The aluminium alloy 2024 properties, such as high strength-to-weight ratio, good fracture toughness and low cost, have maintained its dominant position in aerospace industry [1]. This kind of aluminium alloy has a complex distribution and composition of intermetallic particles. It is well known that corrosion process initiate in intermetallic particles, specifically in the S-phase with preferential dissolution of Al and Mg [2,3]. The localized corrosion constitutes the main degradation form of this alloy [2,4,5]. According to some authors the formation of stable pits evolves the cooperative mechanism, by galvanic coupling of intermetallic particles [6]. The cooperative corrosion process comprise the appearance and development of corrosion

rings, whose intern surface act predominantly as a cathode and drives the anode into the surface, resulting in the grain boundary network. The grain boundaries attack evolves into an intergranular attack, leading to mechanical failure of this aluminium alloy [7].

One of the most important methods for corrosion protection is the employment of organic coatings, such as anticorrosive paints. A key feature in lifetime of organic coatings is its adhesion with the metal surface when exposed to corrosive medium [8], *i.e.*, the adhesion is one of the fundamental properties for a good corrosion protection. The physical–chemistry properties in the interface between metal and paint are usually improved by a pretreatment. The chromatization was a pretreatment widely used for light metals such as aluminium and its alloys, especially in the aerospace industry [9] due to its role as both anchoring for the organic coating and as corrosion inhibiting system. Nevertheless, the environmental restrictions have forbidden its utilization. In this context, the sol–gel process based on silica precursors are able to form bonds with both metal substrate and reactive groups of organic paint thought their functionalized organic group, due to silane bifunctionality [10]. Thus, as silane films assist the interaction and adhesion with ensuing organic coating layer, they have

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been appointed as pretreatment or precoating for further paint deposition. Moreover, during the last years, attention has been given to the development of functional barrier coatings based on the modification of epoxy resins or polyurethanes with the incorporation of silanes or polyaniline molecules [11].

A wide amount of works concerning the anticorrosive performance of metals coated with sol–gel films obtained by silica precursors has been published [12–14]. Nevertheless, studies regarding the employment of sol–gel films as previous nanocoating layer for further paint deposition and metal protection are scarce. As an example, Bajat et al. [15] has described the use of vinylsilane as a pretreatment of aluminium surface before epoxy paint deposition. On the other hand, Zand and Mahdavian [16] also reported the use of sol–gel films as previous coating for polyurethane paint deposition. They obtained good results of adhesion strength and corrosion protection.

The behaviour of sol–gel films based on silica precursors as adhesion promoters and its anticorrosion action has been dependent on parameters such as, concentration of silane in the starting solution [15,17,18], silica precursor nature [17,18], pH of the starting solution [16,17], immersion time in the starting solution [15], nature of organic coatings [18] and the nature of the metal substrate [19].

Moreover, the silica precursor must be chosen so as to guarantee proper compatibility with the polymeric resin and to provide good interaction with the metal substrate. In our previous work we verified that the silica precursors, vinyltrimethoxysilane (VTMS)/tetraethylorthosilicate (TEOS), when mixed in adequate ratio (3/1) propitiate the suitable balance between hydrophobicity and porosity to be used as anticorrosive barrier and adhesion promoter [20]. We also verified that the incorporation of a catalytic amount of phosphonic acid, ethylenediaminetetra(methylene phosphonic acid) (EDTPO), to silica network promoted the formation of Al–O–P bonds, resulting in a great benefit for silane adhesion and corrosion protection [21–25].

Therefore, the main aim of the present work is combine the anticorrosive and adhesion properties propitiated by VTMS/TEOS nanocoating covalently bonded to the aluminium surface with a well-known anticorrosive epoxy primer, forming a bilayer system for the protection of AA2024 aluminium alloy. In order to improve the previous results with AA2024 alloy, two further approaches were investigated: (i) the incorporation of EDTPO to the VTMS/TEOS sol–gel formulation and (ii) the incorporation of a conducting polymer to the epoxy paint. Since the advantage combination of the use of conducting polymers (CPs), as anticorrosive additive, with organic coatings has granted the improvement of corrosion protection of metals [26–30], accordingly, we incorporated a soluble semiconducting polythiophene derivative (P3TMA) to the epoxy paint formulation in order to evaluate its influence to the adhesion between the sol–gel inner nanocoating and the epoxy outer coating.

2. Experimental

2.1. Preparation of AA2024 substrate and sol–gel films deposition

AA2024-T3 rectangular samples 5 × 10 cm were prepared by grinding with silicon carbide papers up to 1200 grit. For the aluminium alloy treated with silica precursors, the samples was firstly immersed in a 0.05 mol L⁻¹ acetic acid solution for 5 min and washed with distilled water and dried under a hot air stream [23]. Then, the panels were immersed in the starting solutions of silica precursors for 30 min, followed by dry in an oven at 110 °C during 1 h for curing. The starting solutions consist of 50% (v/v) ethanol (Nuclear, 99.5%), 46% (v/v) deionized water (18.3 MΩ cm),

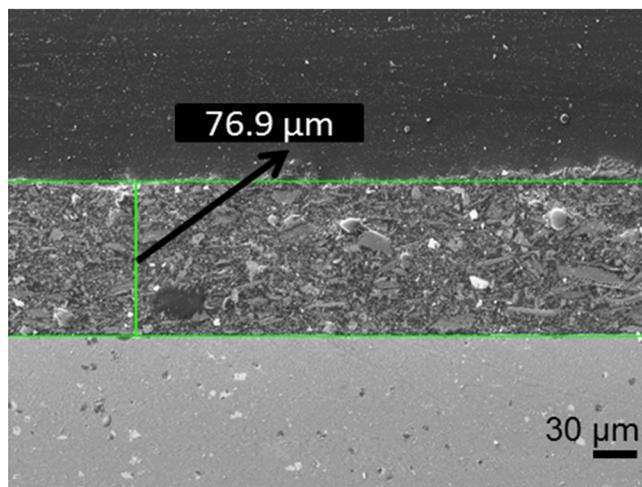


Fig. 1. Cross section scanning electron microscopy of epoxy paint deposited onto AA2024-T3.

1% (v/v) TEOS (Merck, 98%) and 3% (v/v) VTMS (Aldrich, 97%), which were mixed for 1 h and stored for 3 days prior use. The phosphonic acid EDTPO (Dojindo, Japan, 97%) was added to this starting solution with molar concentration of 3.75×10^{-5} mol L⁻¹. The resulting films were denoted as 3V and 3VE5 for sol–gel without and with phosphonic acid, respectively.

The thicknesses of the as-prepared 3V and 3VE5 films were measured with a profilometer Dektack Veeco 150. The sol–gel films thickness varied from 190 to 200 nm, respectively, without and with phosphonic acid adhesion promoter.

2.2. Paint application

The primer applied after the sol–gel films deposited onto AA2024 surface is composed by two components epoxy-polyamide material commercialized by Industrias Titan S.A. (Epoxy bicomponent Titan 831). The resin-hardener ratio (v/v) used was 4/1 according to the manufacturer recommendations. The mixture was prepared at room temperature by stirring for about 20 min before metal painting. The samples coated with sol–gel film were immersed into the reservoir containing the liquid paint. The painted specimens were allowed to cure for 7 days at room temperature. The conducting polymer, poly(3-methyl thiophene acetate) (P3TMA), was synthesized following the procedure described by Kim et al. [31] and was incorporated into commercial epoxy paint in the concentration of 1% wt. dispersed in chloroform solvent. Before addition, the CP powder was sonicated carefully and filtrated to eliminate any insoluble particle, and it was vigorously stirred to good homogenization in the liquid paint.

Once the paint was completely dried, thickness was measured by using an electrometer Mega-Check Pocket (Neurtek S.A.). The apparatus was previously calibrated to non-ferrous substrate by using the gauges supplied. The epoxy coating thickness varied from 70 to 80 μm, in agreement with the thickness estimated by cross-section SEM images (Fig. 1).

2.3. Measurements

2.3.1. Infrared spectroscopy (FTIR)

The composition of the solid films, either sol–gel films or organic coatings, was monitored by FTIR spectroscopy. Structural characterization of the cured silane nanocoating, covalently bonded onto aluminium surface, was performed using a Nicolet 6700 FTIR spectrometer equipped with Smart SAGA (specular aperture grazing angle, with an incidence angle of 80° from the normal surface)

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