ARTICLE IN PRESS

Progress in Organic Coatings xxx (xxxx) xxx–xxx

Contents lists available at ScienceDirect

Progress in Organic Coatings

journal homepage: www.elsevier.com/locate/porgcoat

Electrochemical study of the synergic effect of phosphorus and cerium additions on a sol-gel coating for Titanium manufactured by powder metallurgy

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ARTICLE INFO

Keywords:
Corrosion
Sol-gel
Coatings
EIS
Inhibitors
Titanium

ABSTRACT

The goal of this work was to combine the physical barrier properties provided by the sol-gel network with an active-chemistry protection against corrosion provided by corrosion inhibitors and cross-linking agents. Sol-gel coatings, organic–inorganic hybrid materials were prepared by hydrolysis and condensation of (3-glycidyloxypropyl)trimethoxysilane (GPTMS) and tetraethylorthosilane (TEOS) in an ethanol/water solution. This coatings were satisfactorily modified by adding an inhibitor of the corrosion, namely hexahydrate cerium nitrate, and a cross-linking agent, namely tris(trimethylsilyl)phosphite (TMSP). The synergetic effect of these two compounds was evaluated by means of electrochemical characterization. The evolution rate of the hydrolysis-polycondensation reaction was monitored by Fourier-transform infrared spectroscopy and viscosity. Once the hydrolysis-polycondensation rate was reached, sols were deposited on titanium fabricated by powder metallurgy. The morphological characteristics and elemental distribution of the coatings were studied using scanning electrochemical microscopy (SEM). The solid-state of Si-NMR spectroscopy and thermogravimetric analysis (TGA) was employed in order to identify the cross-linking state of the coating by ensuring the creation of enough siloxane bonds. Impedance measurements were carried out to study the effect of cerium and TMSP on the corrosion resistance. It was observed that the coating with additions of cerium presented stable and good barrier features against 5 mM NaCl solution. The synergy between the cerium inhibitor and the TMSP cross-linking agent was found not to exert a positive effect because the electrochemical behaviour was dominated by the phosphorus compound over the cerium.

1. Introduction

Due to its high strength, low density, excellent corrosion resistance and biocompatibility with the human body, titanium is an attractive structural material considerably used in several sectors such as aeronautical, aerospace, automobile industry and medical applications [1–3]. The main issues concerning the use of titanium are the costs associated with the mineral extraction and the complex procedure to avoid its contamination. Several works are currently developing alternative procedures for titanium. Amherd Hidalgo, A. et al. [4] provided an extended review of how to enhance the sintering step of titanium by alloying titanium with copper, nickel or iron. Neves, R. G. et al. [5] designed powders of titanium by combining a conventional powder metallurgy procedure with colloidal techniques. Due to the strong affinity that Ti has to oxygen, nitrogen and carbon, sintering is performed under high vacuum to avoid the precipitation of intermetallics [4,6,7].

In order to protect, maintain and prolong the properties of metals, hybrid organic-inorganic sol-gel coatings have been source of attention from the past decades [8–10]. This materials represent an environmentally friendly alternative for chromate coatings, which have been forbidden in Europe since 2007 due to their highly carcinogenic effect [11]. Hybrid organic-inorganic coatings can act as a barrier layer to isolate metallic substrates from its environment [12], however, sometimes they fail to protect substrates once the coating is damaged. The challenge in this field is to combine the physical barrier properties provided by the sol-gel network with an active-chemistry protection against corrosion provided by corrosion inhibitors, cross-linking agents or both of this substances [13].

Sol-gel technology is a chemical synthesis process that creates an organic-inorganic coating that can effectively accommodate different protective substances against corrosion for the performance of materials [8,14]. However, introducing this corrosion inhibitors/cross-

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https://doi.org/10.1016/j.porgcoat.2018.01.026
Received 1 September 2017; Received in revised form 26 January 2018; Accepted 26 January 2018
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Table 1
Summary of the sol-gel doped.

Nomenclature	Component	Silanes:component/ molar ratio	wt.% of component
Control	–	–	–
Ce	$Ce(NO_3)_3{\cdot}6H_20$	–	0.2
TMSP	tris(trimethylsilyl) phosphite	200/1	–
Ce + TMSP	$Ce(NO_3)_3{\cdot}6H_20$ + tris (trimethylsilyl) phosphite	200/1	0.2

Table 2
Assignment of FTIR peaks of Fig. 1.

Wavenumber (cm^{-1})	Assignment
3100–3700	υ(O—H)
3000–2900	υ(C—H)
1636	δ(O—H)
1500–1300	δ(C—H)
1250–1000	υ(Si—OH)
	υ(Si—C)
1187	υ(Si—O—Si)
1100–1070	υ (Si—O—C)

linking agents could compromise the stability of the sol-gel matrix, and also create cracks on the coating surface during the drying step. In order to avoid or minimized these issue, the optimization of the sol-gel procedure must be done by modifying different parameters of the procedure (i.e., the weight percentage of the substances added, the synthesis time, the viscosity of the sol prior to deposition, the curing time and temperature).

In this work two alcoxysilanes (GPTMS and TEOS) were used to prepare a sol-gel coating. Two strategies were employed to provide the coating with (1) physical barrier feature by adding a cross-liking agent and (2) active-chemistry protection with a controlled deliver of an inhibitor. Cross-linking agents are compounds that contribute to the formation of the sol-gel network. They can react with the alcoxysilanes and form covalent bonds. Cross-linking agents improve the adhesion between incompatible inorganic surface and/or organic polymers through intra or intermolecular reaction [15]. Several articles [16–18] confirm the improvement on the corrosion protection of magnesium alloys by applying a sol-gel coating doped with tris(trimethylsilyl) phosphite. This improvement on the corrosion protection is associated with the formation of hydrolytically stable Mg—O—P chemical bonds. Concerning inhibitors, a wide range of substances has been employed to inhibit the corrosion of metals such as aluminum [19] and steel [13,30]. An excellent alternative for replacing Cr(VI) consist on the addition of rare earth elements. Lanthanide ions such as Ce^{3+}, Y^{3+}, La^{3+}, Pr^{3+}, Nd^{3+} seem to fulfil the basic requirements as alternative corrosion inhibitors by providing remarkable adhesion promotion, good barrier properties and self-healing properties [16,11,21].

Table 3
Mean viscosity values calculated for each synthesis.

Reaction time (h)	Viscosity (mPa s)			
	Control	Ce	TMSP	Ce + TMSP
1	4.6 ± 0.2	4.3 ± 0.4	4.1 ± 0.2	5.1 ± 0.3
8	4.4 ± 0.4	4.3 ± 0.7	4.9 ± 0.3	4.8 ± 0.3
24	5.6 ± 0.5	5.9 ± 0.3	9.3 ± 0.4	8.0 ± 0.4
48	9.4 ± 0.4	10.9 ± 0.5	25.2 ± 1.2	18.5 ± 0.9
72	40.0 ± 2.0	45.8 ± 2.0	130.8 ± 8.0	57.6 ± 3.0

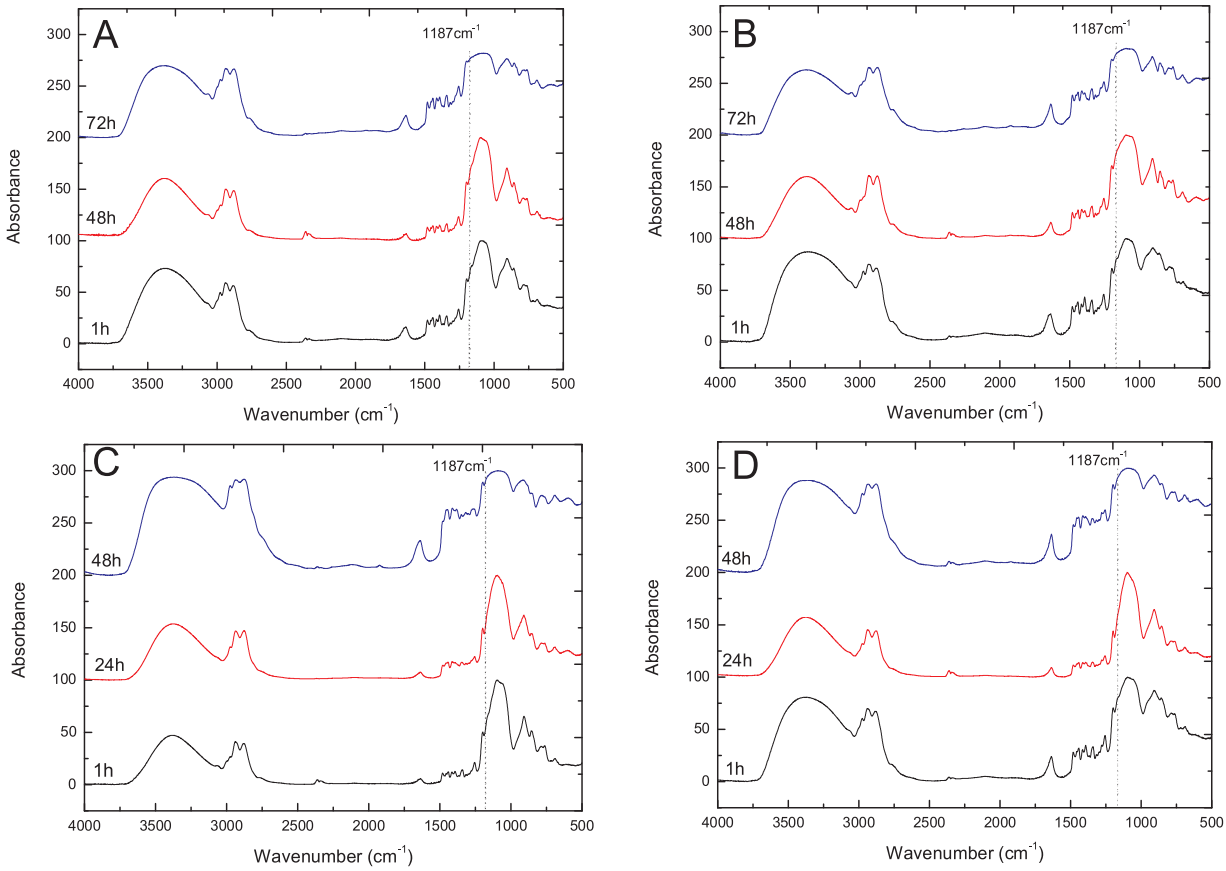

Fig. 1. FTIR spectra of synthesis: (A) Control, (B) Ce, (C) TMSP and (D) Ce + TMSP at different reactions times (from 1 h to 72 h).

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