



# Influence of the sol-gel mesoporosity on the corrosion protection given by an epoxy primer applied on aluminum alloy 2024 –T3

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## ABSTRACT

Silica-based sol-gel mesoporous thin films were applied on aluminum 2024 as first pretreatment surface whereas an epoxy coating was applied as top layer. Non porous and mesoporous sol-gel films were synthesized via EISA process using Pluronic P123 and F108 as templating agents. Two different kinds of alkoxy-silane mixture were compared using inorganic or hybrid precursors. The morphology, the pore size distribution of sol-gel thin films were observed by TEM, FE-SEM and measured by a quartz crystal microbalance (QCM). The anticorrosion performance and characteristics of sol-gel films were assessed by EIS measurements. The compatibility between sol-gel film and epoxy coating was studied by salt spray test and swelling in N-methylpyrrolidone. The goal of this study is to highlight the role of pretreatment porosity at the sol-gel film/organic coating interface on aluminum 2024 substrate. The higher pore size distribution of the hybrid sol-gel formulation promotes adhesion and anticorrosion performance at aluminum 2024 substrate/sol-gel film/epoxy coating interface.

## 1. Introduction

The aluminum alloy 2024 (AA2024) is widely used in aerospace industry due to its low weight and its passive behavior. However, due to the presence of alloying elements such as Cu, Mn, Fe, etc. this alloy is highly sensitive to localized corrosion [1–3]. Generally, chromated-based conversion treatments combined to an epoxy primer are well-known to be very efficient for corrosion protection of AA2024. Unfortunately, due to its effect on human health and environment, the use of hexavalent chromium (VI) and other compounds containing chromium have been limited since 1982 [4] and should be forbidden in aerospace industry in 2024. Among of alternative surface treatments, the sol-gel coating is considered as a friendly-environmental way to avoid the use of chromate in the conversion coatings [4–6].

The sol-gel chemistry is based on the hydrolysis and condensation reactions of precursors, offering a large range of materials used in many applications [6,7]. In recent years, sol-gel coatings have become one of the most promising technologies in corrosion protection of materials. The sol-gel deposit can be applied as the first pre-treatment layer whereas an organic coating deposited onto the silane coating can be considered as the second layer [8–10].

One of the important factors that can guaranty the compatibility between sol-gel layer and epoxy primer is adhesion performance, which

can be achieved by a good pre-treatment surface like sandblasting [11,12], Zr/Ti conversion [13] or anodizing layer [14–16]. It has been reported that pre-treatment based on a sol-gel film can not only improve the corrosion protection of aluminum substrate as a barrier layer but also provide a very good adhesion with the organic coating following the chemical/thermodynamic bonds at substrate/sol-gel layer/organic coating interface [17–21].

In recent years, among of sol-gel technologies, the synthesis of mesoporous sol-gel thin films synthesized via Evaporation Induced Self-Assembly (EISA) [22–24] has shown a high interest for many applications. Compared to the classical sol-gel film deposition, the main difference is the presence of a templating agent in the mixture which can form micelles during the dip-coating due to solvent evaporation. During application of the sol on the substrate, these micelles will change into mesophases due to evaporation of alcohol. This method can allow the formation of ordered mesoporous (2–50 nm) structure with controlled average pore size and pore size distribution [25,26]. The surfactant can be eliminated by calcination or UV/Ozone treatment to obtain mesoporous materials [27,28]. Sol-gel films are known as effective adhesion promoters to enhance the adhesion performance of organic coatings to aluminum substrate [29]. Besides that, the presence of irregularity elements like microcavities or mesopores can also improve the mechanical adhesion [30]. In the present case, the mesoporous sol-gel

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films can combine the advantages of both sol-gel chemistry and mesoporous structure. It was previously shown that the use of a thermal treatment at 350 °C for removing the templating agent and creating the porosity can modify the mechanical properties of 2024 alloy and more particularly its Vickers hardness. Nevertheless, an exposure of an inorganic sol-gel containing P123 to UV/ozone treatment for 24 h can also allow to achieve the same porosity properties without affecting the mechanical properties [31] and could be used instead of the calcination treatment after parameters optimization. As in this study, the focus is given to the influence of the porosity on the corrosion performance and not on the mechanical properties; a thermal calcination treatment will be used to validate the porosity effect.

Sol-gel (SG) thin films having a mesoporous structure has been synthesized via EISA process. P123 and F108 from Pluronic family have been used as two templating agents to synthesized sol-gel films with different pore size distribution. Besides that, the 3-glycidioxypropyltrimethoxysilane (GPTMS) and methyltriethoxysilane (MTES) were added in the mixture to investigate the role of organic precursors on the adhesion performance. The main objective of this study is to highlight the role of pretreatment porosity and sol-gel formulation for the adhesion performance at the sol-gel layer/organic coating interface on aluminum 2024-T3 substrate.

The morphology and the pore size distribution of sol-gel thin films were characterized by TEM and FE-SEM. The porosity and the other characteristic parameters of mesoporous structure (volume, specific area.) were measured by using a quartz crystal microbalance (QCM). The electrochemical impedance spectroscopy was used to confirm the interconnection of pores through the substrate and to evaluate the corrosion protection of AA2024 samples pre-treated and painted with epoxy coatings. The compatibility between sol-gel thin films and epoxy primer was studied by swelling in *n*-methylpyrrolidone and salt spray test.

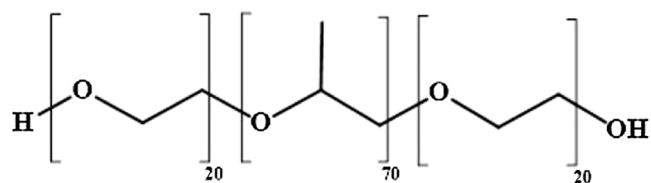
## 2. Experimental

### 2.1. Materials

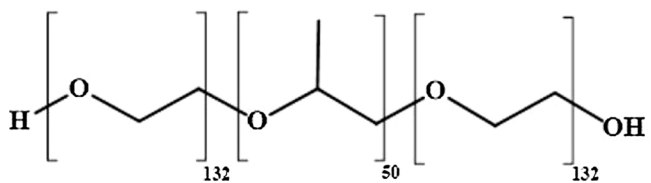
Aluminum alloy AA2024 (provided by Sonaca company, Belgium) panels 4 cm x 6 cm, 1 mm thickness were rinsed with distilled water, acetone and then chemically etched in Turco NC 4215 LT alkaline solution at 50 °C for 10 min. This pretreatment can provide a well-cleaned aluminum substrate and favor the formation of OH groups at the surface, which can improve the adhesion.

Alkoxy-silane mixtures were prepared by using Tetraethylorthosilicate TEOS (Merck, 99%), 3-Glycidioxypropyltrimethoxysilane GPTMS (Sigma Aldrich, 98%), Methyltriethoxysilane MTES (Sigma Aldrich, 98%), concentrated hydrochloric acid (Sigma Aldrich, 32%) and absolute ethanol (Merck, > 99.8%), all chemical products were used as received without further purification.

Pluronic P123 and F108 (Sigma Aldrich) are tri-block hydrophilic-hydrophobic-hydrophilic copolymers with the general formulation (polyethylene)<sub>x</sub>-(polypropylene)<sub>y</sub>-(polyethylene)<sub>x</sub> or (PEO)<sub>x</sub>-(PPO)<sub>y</sub>-(PEO)<sub>x</sub>, as shown in Fig. 1. The pore diameter and the pore size distribution depend significantly on the length of PEO and PPO blocks. The choice of these templating agents is based on multiple reasons [27,28,31–34]. Firstly, among the surfactants, P123 and F108, which are non ionic block copolymers, can create hydrogen bonds interactions with silica matrix, that are weaker than electrostatic and then more easily removed by thermal or ozone treatment. Secondly, mesoporous sol-gel thin films can be achieved with uniform and high ordered pore size. Finally, the P123 and F108 with approximately similar lengths of PPO-chains but different number of PEO-blocks have been used to investigate the influence of hydrophobic-hydrophilic blocks on the pore size distribution



(a) Pluronic P123 : (PEO)<sub>20</sub>-(PPO)<sub>70</sub>-(PEO)<sub>20</sub>



(b) Pluronic F108 : (PEO)<sub>132</sub>-(PPO)<sub>50</sub>-(PEO)<sub>132</sub>

Fig. 1. Chemical structure of (a) Pluronic P123 and (b) Pluronic F108.

Table 1

Molar compositions corresponds to all sol-gel thin films.

List of samples	Abbreviation	Molar composition			
		TEOS	GPTMS	MTES	Templating agent
Aluminum non thermally treated	AL0				
Inorganic non porous film cured at 150 °C	WT1	1			
Inorganic mesoporous film with P123	MP1	1			5 × 10 <sup>-3</sup>
Inorganic mesoporous film with F108	MF1	1			3 × 10 <sup>-3</sup>
Hybrid mesoporous film with F108	MF2	1/3	1/3	1/3	6 × 10 <sup>-3</sup>

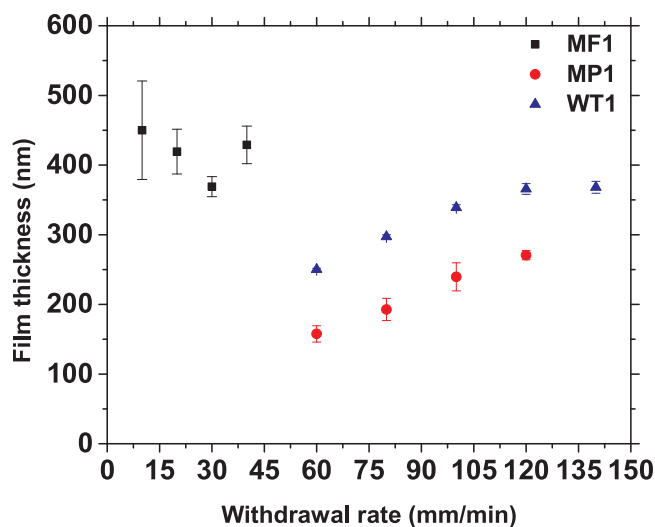


Fig. 2. Influence of withdrawal rate on the film thickness.

### 2.2. Experimental procedure

#### 2.2.1. Sol-gel preparation

The molar compositions corresponding to the mesoporous sol-gel thin films and inorganic non porous sol-gel thin film used as reference were presented in Table 1. pH of solution was maintained at 3–3.5 by using 10<sup>-2</sup> M HCl solution. Experimental parameters required for each

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