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The Influence of Sample Preparation Techniques on Aluminium Alloy AA2024-T3 Substrates for Sol-Gel Coating †

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Abstract: Sol-gel coatings provide environmentally friendly surface protection of metals and can replace toxic pre-treatments such as those based on hexavalent chromium on metal alloys. The project ultimately aims to develop silica-based organic-inorganic sol-gel derived thin film coatings possessing anti-corrosion and anti-fouling properties on aluminium alloys substrates. As with any coating, sample preparation plays a significant role in the performance of a sol-gel coating. Therefore, it was necessary to define a preparation method that combines the removal of contaminants and surface roughening to improve adhesion and reproducibility. Four techniques were investigated: fine abrasive sandpaper cleaning, acetone degreasing only and cleaning with an industrial-available alkaline cleaner for 5 minutes and 30 minutes.

Keywords: Silica-based hybrid sol-gel coating, infrared spectroscopy, electrochemical testing, corrosion protection.

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1. Introduction

Aluminium alloys are still considered one of the primary light alloys with high strength that can be used in aerospace and marine structures with a moderate economic cost. However, depending on the type and grade, aluminium alloys are Vulnerable to aggressive environments[1,2]. Surface pollutants, like organic dirt, grease and lubricants, must be removed to promote ionic or mechanical bonding between coatings and the substrates. Some of the practising procedures focus on degreasing more than removing oxides or cladding film, as it still reduces the corrosion propagation in the surface[3]. The standard aluminium alloys surface preparation in the marine application can be achieved using a high alkaline solvent jet to remove the organic and greasing residual from 5 to 30 minutes, then it could be followed or mixed with sandblasting to remove the oxide film [4].

The hybrid silica-based sol-gel coatings have already been recognised as a potential corrosion mitigation solution for aerospace and marine use as an eco-friendly method [5,6], offering many routes, including using single-or multi-layer coating systems with anti-corrosion and anti-fouling systems. [5,7–9] Additionally, sol-gel coatings can present other desirable properties, such as preventing ice accumulation, oxidation resistance and abrasion resistance [10–12]. However, In sol-gel, the adhesion mechanism with aluminium alloys' surface is different from the other coating types; it is based on (M-O-Si) as strong ionic bonding. Therefore, This paper will study the optimisation of sample preparations techniques of aluminium alloy 2024-t3 for the sol-gel coating that used in the previous project [1], with four types of surface preparation that suit the sol-gel coating technology to provide proper bonding for protection from corrosion.

Experimental Work

The testing and characterisation techniques will be done in two main steps. The first one will examine the treated substrates without the presence of any sol-gel coating by investigating the morphology, appearance, phase construction and XRD analysing techniques. The second step will be after applying the sol-gel coating on four different treated samples and apply an electrochemical impedance spectroscopy preliminary test to evaluate the corrosion mechanism protection within 6 days in a 3.5% NaCl solution to simulate the aggressive environment.

2.1 *Substrate preparation*

Q-panels were supplied by Q-Lab made of aluminium alloy AA2024-t3 with dimensions (102 mm × 25 mm × 1.6 mm) for use as test substrates [13]. The Q-panels samples will be treated in four different surface preparations as following.

1- The acetone was applied on the surface followed with DI on as received Q-panels samples to remove the organic contamination and grease from the surface, leaving the oxide film layer on, and the sample will be labelled as normal cleaning (NC).
2- The second one will follow the same cleaning method by acetone followed by immersing in Super bee® the Alkaline etching solution 10% for 5 minutes as the minimum time in standard and labelled as (5-AC).
3- The third sample preparation will follow the same cleaning method by acetone but immersed in Super bee® the Alkaline etching solution 10% for 30 minutes as the maximum time in standard and labelled as (30-AC).
4- The fourth sample preparation will be treated with Mechanical abrasive sandpaper to reach P1500 directly to remove the top surface and oxides film and labelled as (SP-C).

2.2 *Sol-Gel preparation*

The hybrid silica-based sol-gel was prepared by mixing tetraethylorthosilicate silane (TEOS), trimethoxymethyl silane (MTMS), and isopropanol (all purchased from Sigma-Aldrich) and with dropwise additions of DI water at the molar ratio of 18: 14: 17: 220, respectively as it mentioned in the previous work [1]. The formula was applied by spray coating on the clean substrate with built up over three passes. After that, the coated samples were left in the atmosphere for 10 min before being thermally annealed at 80° C for 4 hours—the chosen samples with a thickness of 16 ±2 micrometre were chosen.

3. Results and Discussion

3.1 Surface analysis for the uncoated substrates

3.1.1 Infinity focus microscopy IFM

The IFM images in figure 1 show the difference between the four samples preparation techniques. It shows that the surface of the 30 minutes etched sample is affected by aggressive pitting due to the dissolving of the light metals in the alkaline solution. While the sandpaper P1500 finishing sample preparation shows that the surface is very smooth. The appearance of standard cleaning NC samples and 5 minutes etching cleaning is smooth, and the preparations are not affecting the roughness of the surface Rz, which was about 1.2 μm; the highest roughness Rz was 3.3 μm for the 30-AC sample while the smallest was 659nm for the sandpaper cleaning SP-C.

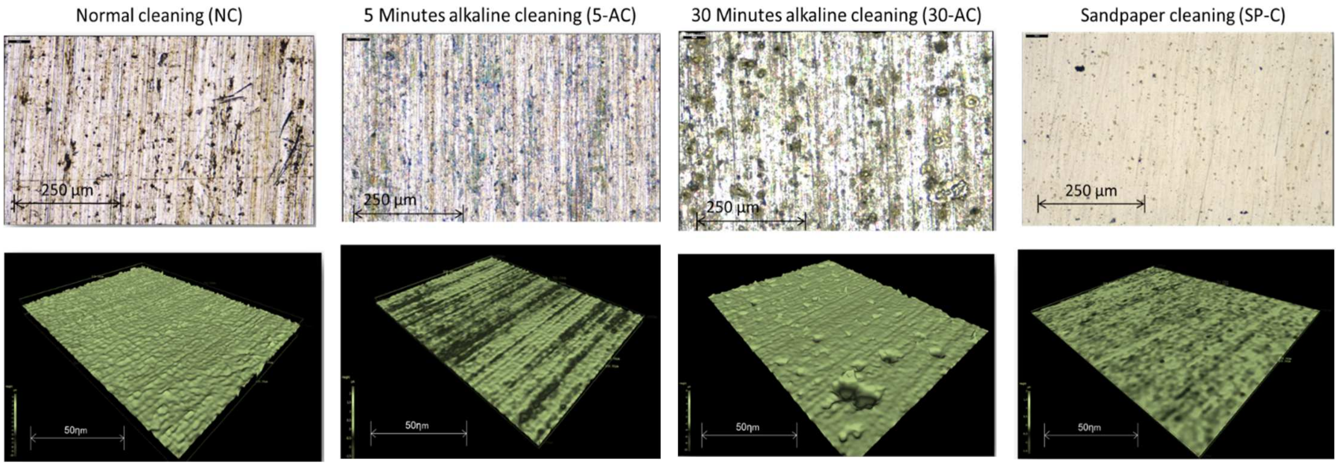

Figure 1, IFM images shows the profile of the four surface preparations samples

3.1.2 Scanning electron microscopy (SEM)

The SEM images in figure 2 show the fours sample preparations' morphological surface appearance as follows: the most affected surface is by 30 minutes etching cleaning. The other surface preparation samples show that the surface is not significantly affected. However, the inclusions from the silicate carbides are impeded in the sandpaper sample SP-C preparations' surface as a result of tribology and friction.

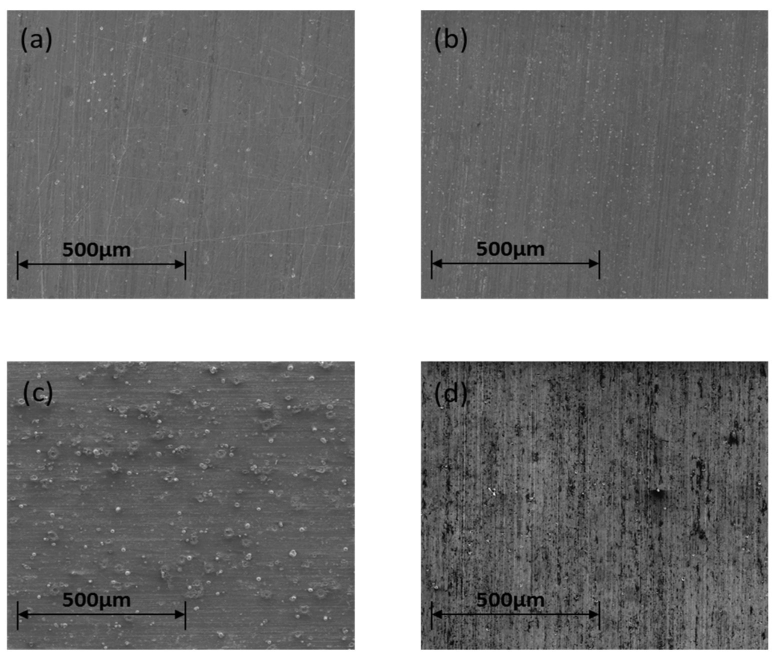

Figure 2, SEM images, Shows the surface for (a) NC, (b) 5-AC, (c) 30-AC and (d) SP-C surface preparations samples

3.1.3 X-ray Powder Diffraction (XRD)

Generally, figure 3 shows there was no significant phase transformation on the surface of the alloy for the four different sample preparations. However, the sandpaper shows fewer aluminium-oxides Al_2O_3 at 38.4°, which show the level of the oxide on the surface is superficial. At the same time, both alkaline etched samples show a higher level of it.

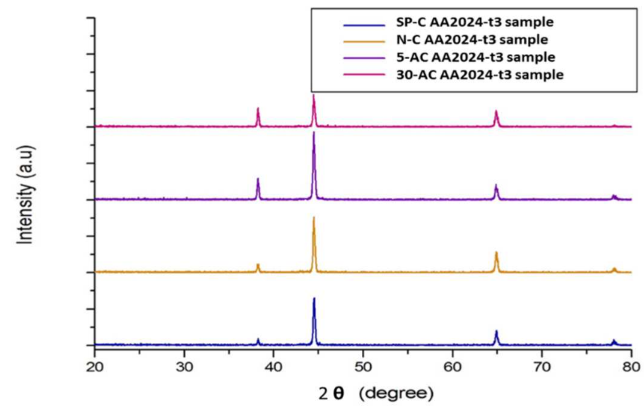

Figure 3, X-ray Diffraction (XRD) reading for the four preparations samples

3.1.4 Wettability and contact angle (WCA)

figure 4 shows the droplets of 10 µl of DI water on the surface of the differently treated samples. It is clear that the highest contact angle was for the 30-AC sample while the smallest was on the sandpaper cleaned sample by 92° and 62°, respectively, which means the 30-AC cleaned sample is less-waitable compared to the others. The other samples of 5-AC and NC samples were 72° and 78°, respectively.

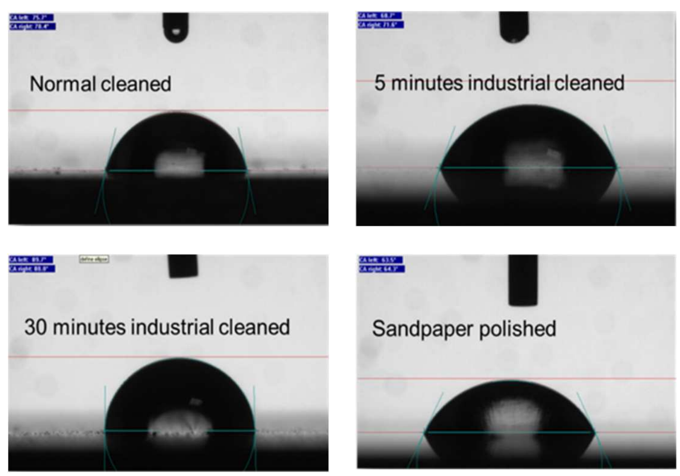

Figure 4, water contact angle for NC, SP-C, 5-AC, and 30-AC sample

3.2 Testing characterisations of sol-gel coated samples

3.2.1 Electrochemical impedance spectroscopy(EIS)

Tests were performed over a period of 6 days. Figures 5 (a) and (b) show Bode impedance magnitude plots for Sol-gel-NC, Sol-gel-SP-C, Sol-gel-5-AC, and Sol-gel-30-AC coated samples in first and after 144 hrs. From Figure 6, The overall impedance for the sol-gel coated samples in the first hour was almost not variant between $5.2X10^6$ to $3.2X10^6$ Ohms/cm^2. The maximum reading was with sol-gel-SP-C, and the minimum was for the Sol-gel-30-AC; the reading for Sol-gel-5-AC and Sol-gel-NC was $4.5X10^6$ and $4.02X10^6$ ohms/cm^2, respectively. After 144 hours, the drop in impedance was clearly apparent by approximately one order of magnitude for the Sol-gel-30-AC sample compared to the Sol-gel-SP-C sample. This might be due to the microcracks in the sol-gel coating on the 30 minutes etched samples and also to the oxides film that reduces the adhesion of the sol-gel film on the surface.

On the other hand, the significance reading of the Sol-gel-5-AC was showed seemlier behaviour to the sandpaper prepared coated sample but with a slight drop in impedance to reach 1.01X10^6 ohms/cm^2 followed with the standard cleaning (degreasing only).

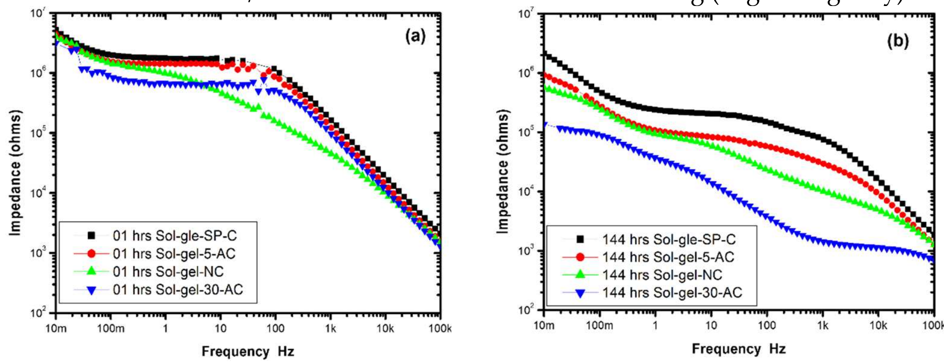

Figure 5, Bode Impedance magnitude plots for Sol-gel-NC, Sol-gel-SP-C, Sol-gel-5-AC, and Sol-gel-30-AC coated samples in the first hour (a) and after 144 hrs (b)

Figures 6 (a) and (b) showed the Nyquist plotting for the four sol-gel coated samples in the 24 hrs, and after 144 hrs, all Nyquist plots support what was mentioned in the Bode plots as they showed one time-constant capacitive behaviour with Wurbirg diffusion element. However, the maximum impedance was for the sol-gel-SP-C coated sample, while the minimum was for Sol-gel-30-AC.

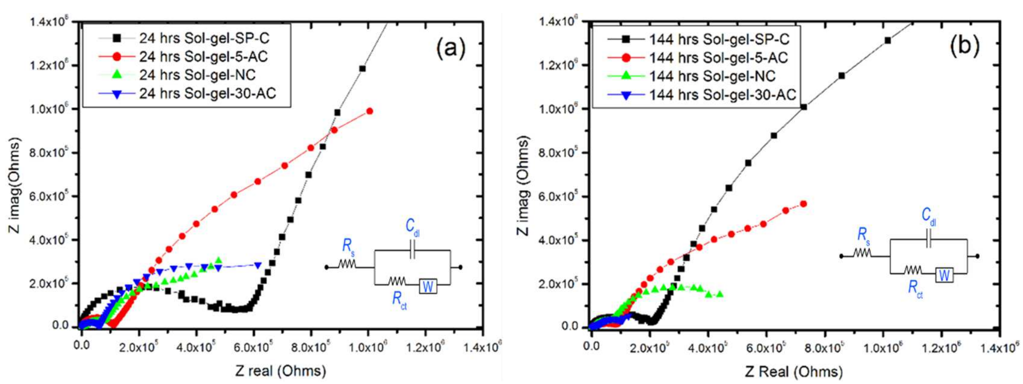

Figure 6, Nyquist plots for Sol-gel-NC, Sol-gel-SP-C, Sol-gel-5-AC, and Sol-gel-30-AC coated samples in 24 hrs(a) and after 144 hrs(b)

4. Conclusions

Mechanical cleaning using abrasive paper produces a smooth surface with good wettability and adhesion for sol-gel. However, there are probably abrasive particles embedded in the surface, which may cause in-future corrosion to occur. Longer immersion times (30 minutes) in 10% Super bee® appear to be less optimal as wettability is decreased, possibly due to excessive surface roughening, while the more active surface results in greater oxidation. On the other hand, 5 minutes immersion in 10% Super bee® gives good wettability and a degree of surface roughening, which may benefit coating adhesion, as well as degreasing process only. Generally, the sol-gel coating can provide excellent prior protection against corrosion for these mentioned samples depending on the way of the surface cleaning process; it is clear from the electrochemical testing that the mechanical sandpaper cleaning was a good combination offered excellent protection. However, due to the time and cost of this process, the 5 minutes of etching cleaning was acceptable for sample preparation for Sol-gel, then comes degreasing only.

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