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Research article

Effect of pretreatments on electrodeposited epoxy coatings for electronic industries

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Abstract: Waterborne epoxy coatings were prepared on aluminium (Al) surfaces by cathodic electro-deposition on the pretreated surface of pickling, phosphating, chromating and anodizing. The electro-deposition experiments were done at two different voltages, 15 V and 25 V at room temperature in 10% epoxy coating formulations. Corrosion and thermal behavior of these coatings were investigated using electrochemical impedance spectroscopy (EIS) and thermo gravimetric analysis (TGA). The coating exhibits better corrosion resistance in anodized Al surface than the other. But, TGA studies show that the thermal stability is higher in anodized and chromated Al surfaces. The surface morphology of these coatings were analyzed by SEM and AFM studies.

Keywords: electrodeposition; A.C impedance; TGA; anodized aluminium; phosphating; chromating

1. Introduction

Aluminum and its alloys have attracted considerable attention due to their wide use in the architectural, aeronautic, automobile and electronic industries. In Automobile industries, the metal surface is coated by a conversion coating, phosphating or chromating to enhance the adhesion and corrosion protection of the substrate. Then, an electrodeposited (ED) coating has been introduced for its excellent corrosion resistance, pore free and uniform coating [1]. The adhesion of an automotive

coating to the metal substrate is depending on both the chemical and physical characteristics of the coating and the substrate.

The chromate based conversion coatings are widely used as pre-treatment for aluminium and Galvanized steel surface due to its superior corrosion resistance and good paint adhesion.This chemical treatment contains soluble chromate (VI) species which are responsible for the self-healing behavior.However,this chromate coating consists of carcinogenic materials. Therefore,environment friendly Chrome (III) coatings are used as an alternate to improve the adhesion of the coating (2). However, this coatings performance are inferior to chrome (VI) coatings [3]. The phosphating has been used as most common surface pretreatment for both ferrous and non-ferrous metals. It has been shown that this pretreatment plays a vital part in the automobile, appliance and general manufacturing industries[4]. Phosphate conversion coatings are usually porous and requires an additional sealing procedure and carried out at higher temperature which requires high energy.

Aluminum is anodized for corrosion resistance,aesthetic appearance, hardness & wear resistance. Further it has number of specialized properties like better adhesion for surface coatings and enhanced capacitance in electronic applications. The pretreatment of high strength aluminium alloys in the aircraft industry traditionally consists of an anodization layer or a conversion coating. In some cases the processes still rely on the use of chromates. The coating system used for aeronautical applications is quite complex and is constituted by several functional layers such as pretreatment, primer and topcoat. It is well known that the natural oxide film present on aluminium is responsible for higher corrosion resistance [5]. The surface pretreatments carried out for the metallic substrates can have a more enhanced lifetime of a metal/ coating system with respect to the protection of aluminium and its alloys [6].

Protective coatings were formed during cathodic electrodeposition of an epoxy resin modified by amine and iso cyanate on aluminium and modified aluminium using constant high voltage method [7]. Electrodeposited epoxy coatings at higher voltages have high values of pore resistance, R_p and low values of coating capacitance,C_c than coatings formed at lower voltages [8]. But the electrodeposition formed at low voltages is highly utilized in the field of high quality printed circuit boards(PCB) production. Considering the essential requirements for fabrication of PCB, epoxy resin is most frequently used as the primer in the electronic industry [9]. Electrodeposition of epoxy polymer coating is a complex process. Epoxy polymers containing double bonds, which are capable of auto-oxidation, can be thermo cured at low temperatures [10].

The present work investigates the low voltage electrodeposited epoxy coating on phosphated, chromated, anodized and pickled aluminium substrate to obtain pore free with enhanced adhesion of organic moiety. The surface morphology studies of pretreated aluminium surface were also carried out to confirm the adhesion properties to improve the protective performance of the coatings.

2. Materials and Method

2.1. Materials

In this study, the chemicals used for phosphating and chromating baths were supplied by Qualikem Fine Chemicals, India Pvt. Ltd. Liquid epoxy resin (GY250) used for cathodic electro

deposition having the epoxy equivalent of 183–189; diethanolamine, propylene glycol and lactic acid were obtained from E Merck.

Aluminium panels of size 7.5 cm × 5.0 cm × 0.1 cm were employed in the experimental process. The panels were degreased with trichloroethylene, followed by alkaline cleaning in a mixture of 2% NaOH and 5% sodium carbonate at room temperature for one minute. After alkaline cleaning, the samples were pickled in 10% HNO_3 for 30 seconds, rinsed in deionized water and dried as per the ASTM standards [11]. After cleaning the surface with distilled water, the samples were pretreated in the following baths:

1. The composition of the phosphate bath comprises of 7 g of zinc oxide dissolved in 20 ml of phosphoric acid and diluted with deionized water. To this solution, add 5 g of sodium nitrate, 0.8 g of sodium fluoride, 0.54 g of hydroxylamine sulfate and 0.6 g of cerium nitrate, stirr well until completely dissolved in water and made up to 1 liter.

2. The composition of the chromate bath contains 23 g of sodium dichromate dissolved in deionized water, add 6 ml of nitric acid and 0.5 g of sodium fluoride, stirr well until completely dissolved in water and made up to 1 liter.

3. The aluminium specimen was anodized in 10% H_2SO_4, at temperatures between 288°K and 293°K, with cell voltage of 10–15 V and current density of 1 Adm^{-2} for 15 minutes.

2.2. Preparation of water soluble epoxy compositions for electrodeposition

Water soluble epoxy compositions for electrodeposition were prepared by mixing 60 g of liquid epoxy, 15 g of propylene glycol and 9 g of diethanolamine in a 500 ml two necked R.B flask connected to a reflux condenser, and the contents were heated to 453°K for 1 hour. The contents were cooled at room temperature, and acidified with 7.25 g of lactic acid, drop by drop till a pH of 6.0–6.5 was attained. The required volume of resin was prepared by mixing with water in the ratio of 1:9.

The electrodeposition was carried out cathodically at two different potentials viz. 15 V and 25 V on aluminium surfaces pretreated by different methods (phosphated, chromated, anodized and pickled). The temperature was kept constant at 303 K and pH at 6.0 with a coating duration of 3 minutes. After coating, the samples were rinsed with water and cured at 353 K for 30 minutes. Film thickness obtained on the aluminium specimens are reported as a function of electro deposition potential in Table 1.

Table 1. Thickness of electrodeposited epoxy coating on aluminum surface.

Potentials,V	Thickness,μm			
	Phosphated	Pickled	Chromated	Anodized
15	8.2	7.5	5.0	12
25	10.2	13.6	7.5	22

2.3. Characterization

The electrochemical properties of the epoxy coating on aluminium and modified aluminium surfaces were determined using Electrochemical Impedance Spectroscopy (EIS). The coated samples were exposed to 3% NaCl solution for 7 days. A three electrode cell arrangement was used. The working electrode was coated panel kept in a special Teflon holder. The counter electrode was platinum, with a surface area considerably greater than that of the working electrode. The reference electrode was a saturated calomel electrode (SCE). A.C. Impedance data were obtained at the open-circuit potential using a PARSTAT 2273 model impedance analyzer. The impedance analysis was carried out over a frequency range of 100 mHz to 100 KHz using 5 mV A.C amplitude of sinusoidal voltage.

Thermogravimetric analysis (TGA) was carried out using a Q600 V8.3 instrument. The experiments were performed in a dynamic nitrogen atmosphere (30 cm^3 min^{-1}) at a heating rate of 283 K min^{-1} over the temperature range of 293–873 K. The surface morphology and profiles of the epoxy coatings obtained at 15 V potentials were examined using SEM (model TESCON VEGA3) and AFM (model PICOSPM Picoscan2100).

3. Results and Discussion

3.1. Electrochemical Measurements

An equivalent electrical circuit model is shown in Fig.1 for the behavior of polymer coated metal in 3% NaCl solution, where R_Ω is the resistance of the electrolyte, R_P is the coating pore resistance due to diffusion of electrolyte, C_c is the coating capacitance, R_{ct} is the charge transfer resistance and Z_w is the Warburg impedance.
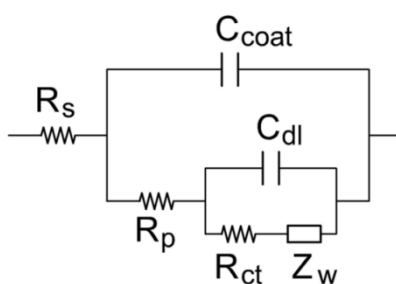

Figure1. Equivalent electrical circuit of a polymer coated metal.

Figures 2–5 show the bode plot of electrodeposited epoxy coating on pretreated aluminum surface. The resistance and capacitance values derived from the Bode plots are given in Table-2.

The impedance diagram (Figure 2) indicates that the Bode plots are inclined to nearly 60^0 towards the X axis of higher frequency. This is the indication of the capacitive behavior of the coating on anodized surface [12,13]. The resistance produced by the coatings is in the order of

10^7 Ω. cm^2. The high resistance value indicates the protective nature of the coating [14]. It is observed from

Table 2. EIS measurements of electrodeposited epoxy coating on pretreated aluminium substrates.

| Epoxy coating on pretreated Aluminium	Voltage	Charge transfer resistance,R_{ct} (Ω. cm^2)			Double layer capacitance ,C_{dl} (F cm^{-2})		
		1 h	1 day	7 days	1 h	1 day	7 days
Anodized	15V	7.998×10^9	4.097×10^{11}	3.013×10^{10}	1.079×10^{-10}	2.153×10^{-11}	1.738×10^{-11}
	25V	3.152×10^9	2.586×10^5	5.159×10^7	3.248×10^{-11}	8.093×10^{-6}	5.954×10^{-11}
chromated	15V	1.483×10^6	1.916×10^5	8.054×10^4	9.701×10^{-8}	1.618×10^{-6}	2.394×10^{-7}
	25V	2.815×10^5	3.35×10^4	3.857×10^5	1.037×10^{-6}	3.257×10^{-5}	9.124×10^{-6}
phosphated	15V	1.092×10^9	6.938×10^4	1.328×10^5	3.651×10^{-11}	1.224×10^{-6}	1.181×10^{-5}
	25V	7399	5.949×10^4	6.394×10^4	1.857×10^{-6}	6.651×10^{-7}	2.499×10^{-7}
Pickled	15V	2.087×10^5	1.319×10^5	5.885×10^4	2.67×10^{-7}	2.015×10^{-7}	7.248×10^{-5}
	25V	1.719×10^5	767.1	577	1.253×10^{-7}	2.769×10^{-4}	3.031×10^{-8}
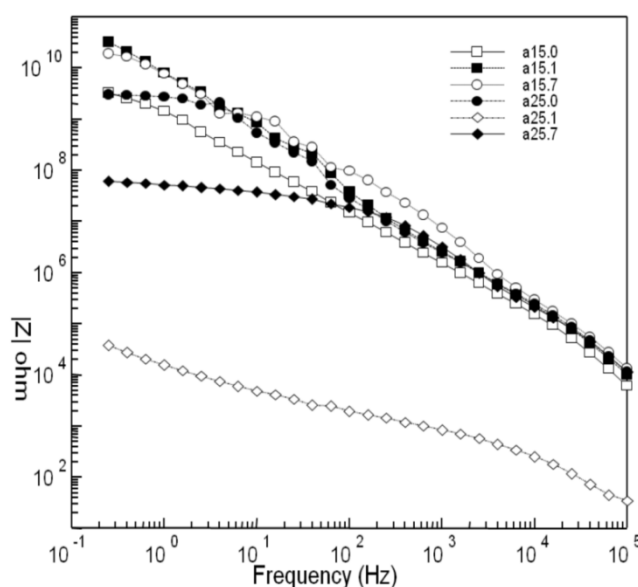
Figure 2. EIS of electrodeposited epoxy coating on anodized aluminium in 3 wt% NaCl Solution with different duration.

table-2, after 7 days of immersion in 3% NaCl, the coating deposited at 15 V exerted higher resistance than the coatings deposited at 25 V. This can be ascribed to the difference in the porosity levels of the epoxy coatings formed on anodized surface at 15 V and 25 V. The higher resistance value of coating formed at 15 V clearly indicates that the coating has low porosity and excellent

adhesion than the coating formed at 25 V. The double layer capacitance (C_{dl}) of epoxy deposited coatings on anodized aluminium for 7days of immersion are, 1.738×10^{-11} and 5.954×10^{-11} F.cm^2 for 15 V and 25 V, respectively. The low values of Cdl and high values of R_{ct} for the epoxy coatings on anodized surface have greater corrosion resistance due to the existence of the passive anodic oxide layer as well as the highly adhesive epoxy coatings on the surface [15]. Such behavior can be explained by the fact that the deposition of epoxy coating at low potential is uniform and have better adhesion than the coating formed at high potential [16].

From the Figure 3, it is observed that the Bode plots are inclined to 30^0 towards the X axis and exerted very low resistance in the order of $10^5 \ \Omega$ cm^2 for 15 V and $10^4 \ \Omega$ cm^2 for 25 V deposited coating for 7 days of immersion in 3% NaCl solution. This indicates that the epoxy coatings deposited at different potential have good adhesion on the phosphated surface. But the epoxy coatings deposited both at 15 V and 25 V, exhibit the value of coating resistance below$10^6 \ \Omega$. cm^2, which indicate that the performance of the coating was below the protective level .Faster hydrogen evolution and lower wettability on phosphated aluminium produce more vacancies in the polymer matrix and causes a more porous structure of the epoxy coating.This explains the lower protective properties of these coatings having smaller values of R_p and R_{ct} and higher values of C_c and C_{dl} [17]. This is well supported by the higher C_{dl} values exerted by the coatings deposited by 15V and 25V, 1.181×10^{-5} and 2.499×10^{-7} F.cm^{-2}, respectively.
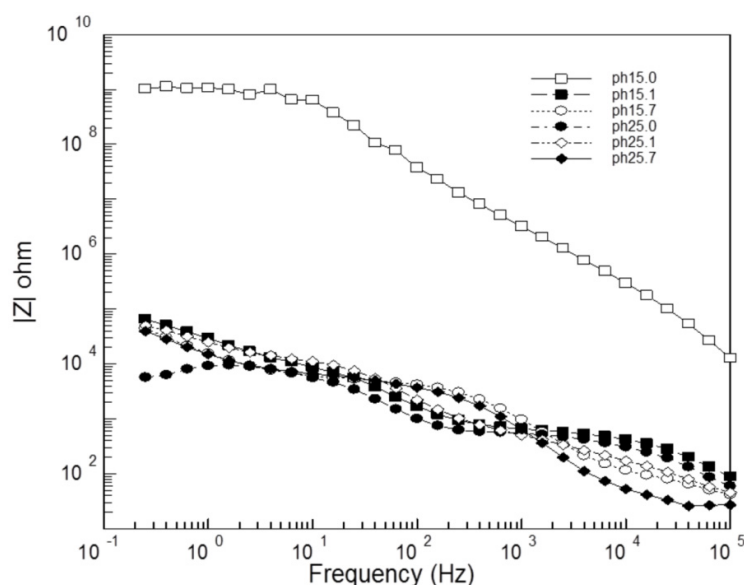

Figure 3. EIS of electrodeposited epoxy coating on phosphated aluminium in 3% NaCl Solution with different duration**.**

From Figure 4, it is observed that the Bode plots behave like the plots obtained with the phosphated surface.The resistance values of epoxy coating produced on chromated Al surfaces after 7 days of immersion in 3% NaCl solution are in the order of $10^4 \ \Omega$ .cm^2 for 15 V, whereas the resistance value of the coating produced at 25 V is $3.857 \times 10^5 \ \Omega$.cm^2 .The protective performance is similar to the phosphated aluminium surface.But the protective mechanism of this chromate surface is due to the formation of chromate passive layer,which reduced Cr^{6+} to Cr^{3+} on the Al surface for the

formation of Cr^{3+} hydroxide monolayer which protects the substrate from the sodium chloride electrolyte [18].
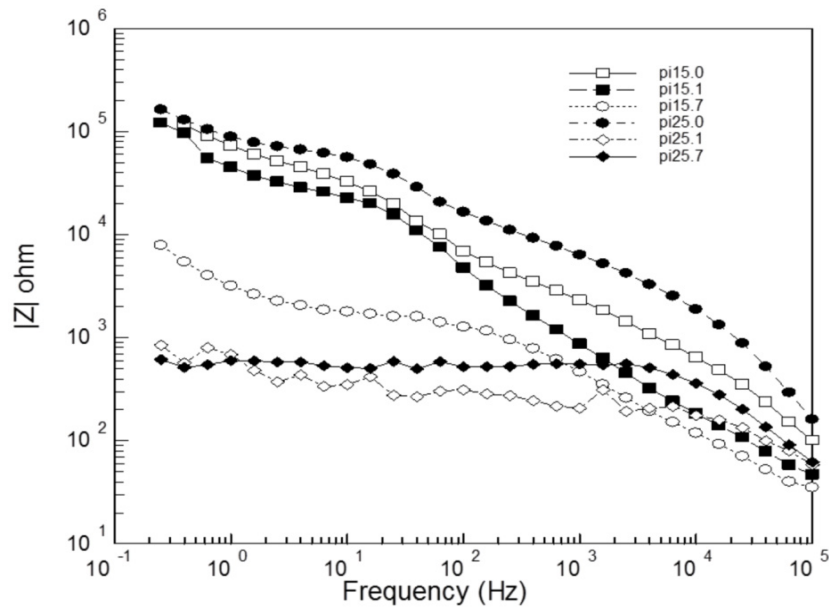

Figure 4. EIS of electrodeposited epoxy coating on chromated aluminium in 3% NaCl Solution with different duration**.**
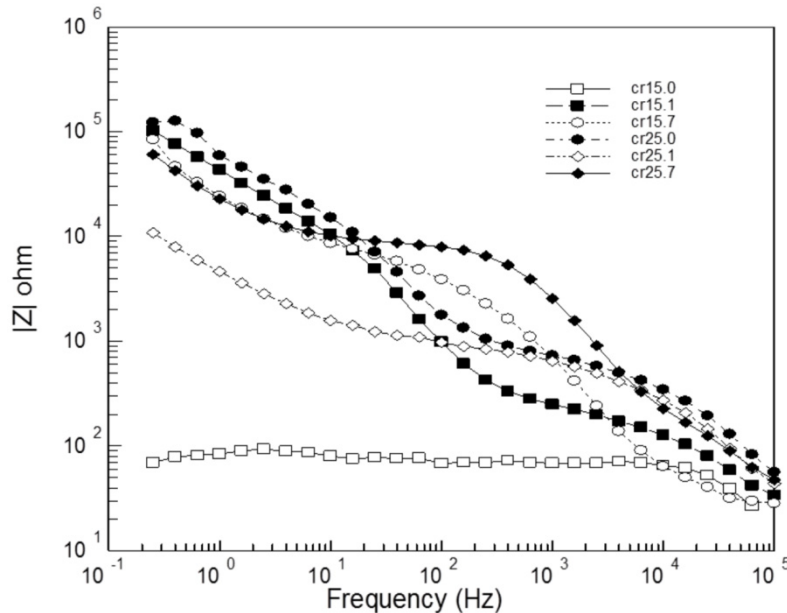

Figure 5. EIS of electrodeposited of epoxy coating on pickled aluminium in 3% NaCl Solution with different duration.

Bode plot obtained from the EIS measurement of electrodeposited epoxy coating on pickled aluminium surface is given in Figure 5. The resistance value of electrodeposited epoxy coatings produced on pickled Al surface, after 7 days of immersion in 3 wt% NaCl, at 15 V, is in the order of

$5.885 \times 10^4 \, \Omega.cm^2$, whereas the resistance value of the coating produced at 25 V is $577 \, \Omega.cm^2$. This could be explained by the formation of a highly porous nature of the coating at 25 V and pore free coating at 15 V. Thus the study shows that the coating formed at 15 V has enhanced corrosion resistance.

3.2. Thermogravimetric Analysis

Thermal stability of high polymers depend on many factors, such as the degree of cross-linking, molecular weight, stability of side substituents, amount of polar groups, intermolecular forces (dipoles and hydrogen bonds), and degree of branching etc. Besides, thermal stability of epoxy polymer depends on the number of benzene rings. The tendency of a polymer to decompose on heating, as a measure of thermal stability of polymer is expressed by three characteristic values
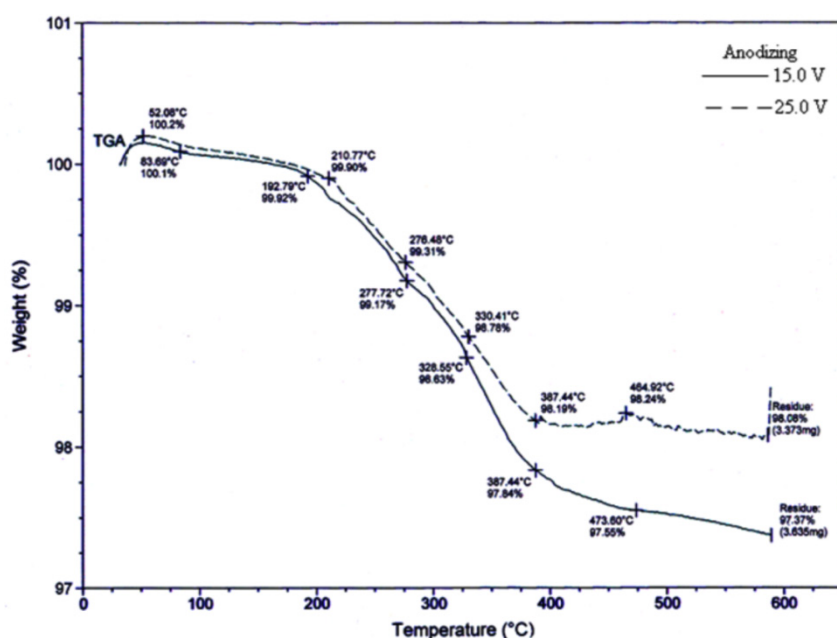
Figure 6. TGA of electrodeposited epoxy coating on anodized aluminium surface at 15 V and 25 V.

Figure 6 shows the TGA patterns of the epoxy coatings obtained on anodized Al surface at 15 V and 25 V. Initially, the weight loss was very low for epoxy coating on anodized Al surface for both 15 V and 25 V, at the temperature of 465.79 K. This is mainly due to the evaporation of moisture and low molecular weight polymers in the coating system. The temperature range between 483.77 K to 660.4 K, the epoxy coating was disintegrated and gave rise to a weight loss value of 2.16% for 15 V, and 1.81% for 25 V. Further the weight loss is minimum up to the temperature 853 K owing to the fact that the anodized aluminium surface is not affected.This can be attributed to the passive Al oxide layer on the surface [19].

The TGA patterns obtained on epoxy coatings deposited on the phosphate pretreated Al surface are shown in Figure 7. At the initial stage, the weight loss was 1.15% for the coating deposited at 15 V at 488.11 K, whereas for the coating deposited at 25 V has no significant indication of weight

loss. This is attributed to the low water content in the coating matrix. At the temperature range 475.59 K to 654.86 K, the epoxy coating was disintegrated and gave rise to a weight loss value of 3.14% for 15 V and 2.76% for 25 V.Thereafter, at 853 K, the weight loss observed was 4.00% and 1.91% for 15 V and 25 V, respectively. This is due to the decomposition of phosphate film on the Al surface [20].
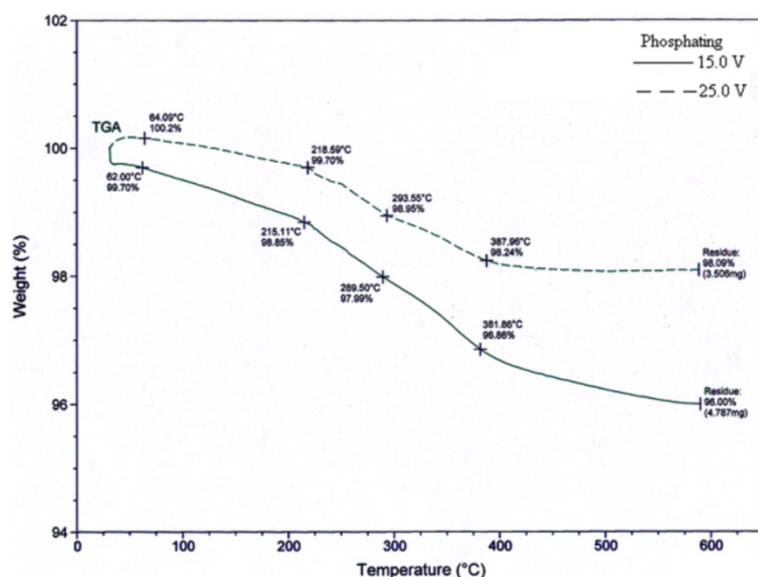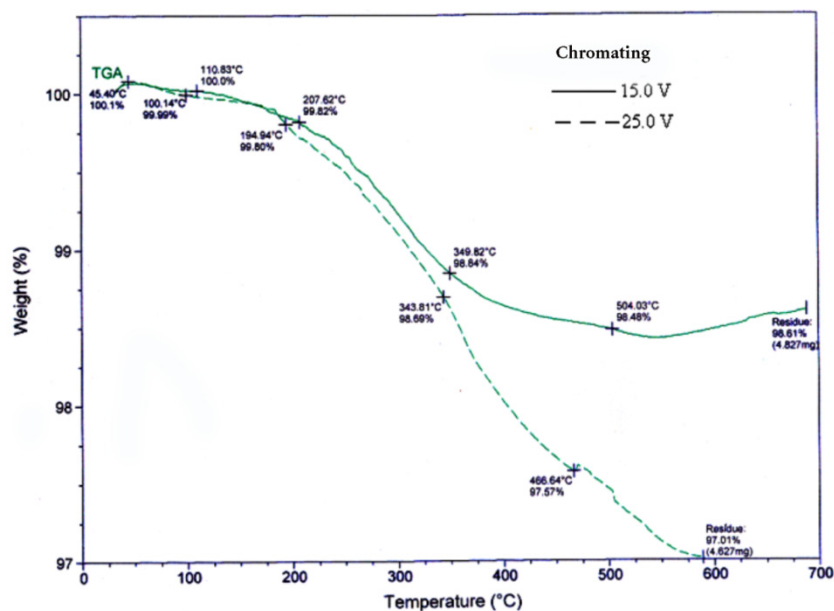

Figure 7. TGA curve of electrodeposited epoxy coating on phosphated aluminium surface at 15 V and 25 V.

Figure 8. TGA curve of electrodeposited epoxy coating on chromated aluminium surface at 15 V and 25 V.

Figure 8 shows TGA patterns obtained on the epoxy coatings electrodeposited on the chromated Al surface at 15 V and 25 V. Initial weight loss of 0.18% and 0.50% were observed for the 15 V and 25 V at 467.94 K, respectively. This is due to low water content and less porous nature of the epoxy coatings on chromated Al surfaces. At 622.82 K, for 15 V potential, the organic moiety in the coating was disintegrated and gave a weight loss of 1.16%, whereas for 25 V, the weight loss was 2.31% at 636.26 K. This can be ascribed to the loss of adhesion of the coatings with respect to the applied deposition voltage. The degradation observed for the epoxy coating on the Al surface deposited at 25 V is found to be linear with a steep fall in the weight loss up to 953 K, whereas it is stable with low weight loss after 873 K for the coating at 15 V. This is attributed to the formation of chromium-chromate mixed oxide as an intermediate layer between metal and epoxy coating on the Al surface [21].
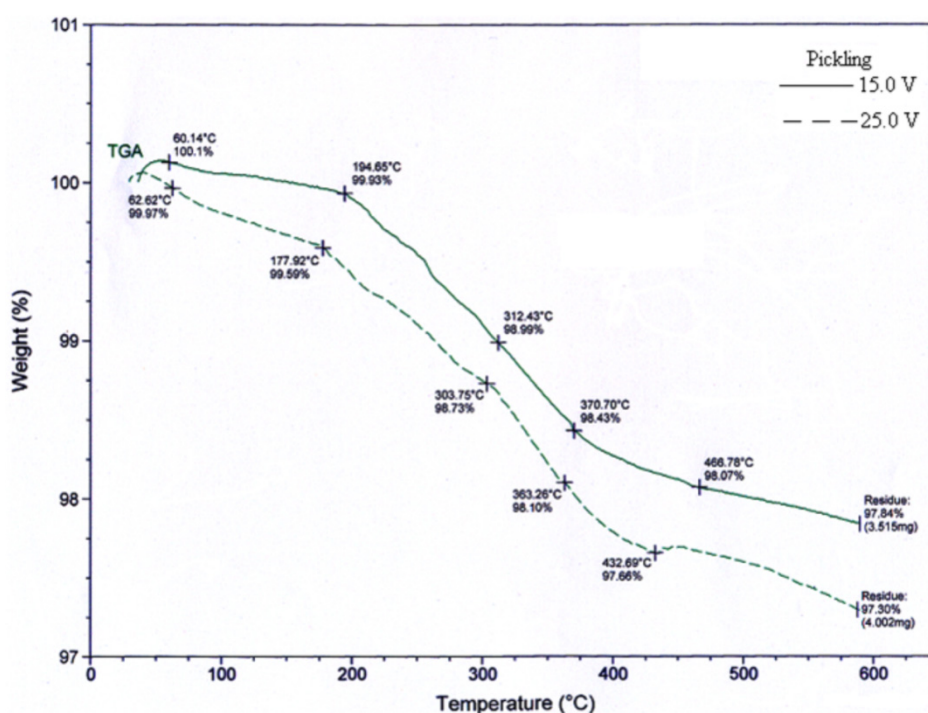
Figure 9. TGA curve of electrodeposited epoxy coating on pickled aluminium surface at 15 V and 25 V.

TGA curves of the epoxy coating deposited on the pickled Al surface at 15 V and 25 V are shown in Figure 9. Up to 467.65 K, the weight loss observed is minimum due to the loss of moisture,impurities and low molecular weight fractions in the coating. Afterwards, the weight steadily decreased and reached a steady state at 873 K,which is mainly due to continuous disintegration of the coating owing to the absence of conversion coating and secondly owing to the poor adhesion and more porous structure of the epoxy coating on pickled Al surface.

This thermogravimetric analysis reveals that the epoxy coating on anodized aluminium surface performed well than the other systems due to the thermal stability of the anodized aluminium. The thermal stability of the epoxy coating on the pickled surface is only depends on the epoxy polymer and so it decomposes steadily with increase in temperature. The phosphating and chromating

pretreatment processes, increase the adhesion of the epoxy coating and so the rate of decomposition is slow and reaches the steady state with the loss in weight of the epoxy coating.

3.3. SEM Studies

The SEM image of the anodized Al surface is shown in Figure 10. It is observed that the oxide film formed on the Al surface is porous in nature.
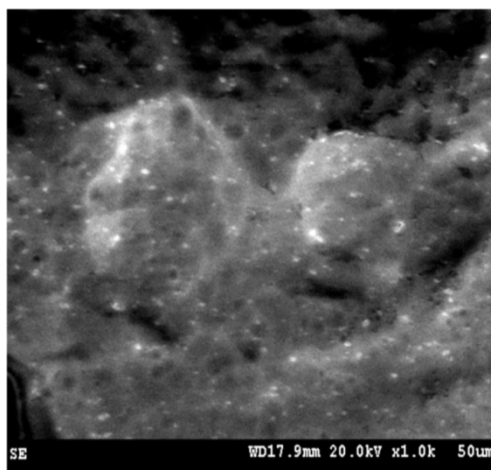

Figure 10. SEM of anodized aluminium surface.

The SEM image of phosphate deposited Al surface is shown in Figure 11.The surface is more porous in nature and sponge like particles. At the longer immersion time, the sponge like particles transformed into distinct crystals and grew rapidly, which indicates that the sponge like particles are the precursors for phosphate crystals.
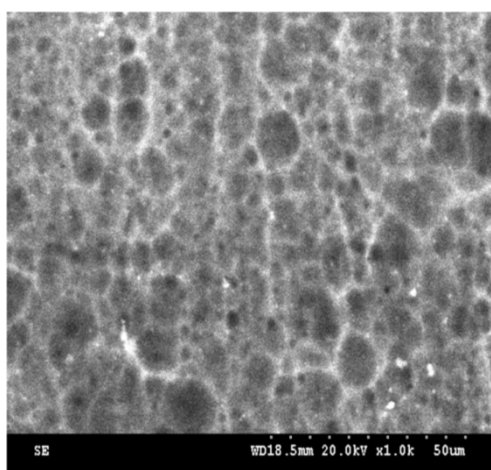
Figure 11. SEM of phosphated aluminium surface.

Figure 12 shows the SEM image on the chromate converted Al surface, having micro-cracks throughout the surface. The appearance of micro-cracks on the surface is due to the low deposition thickness of chromate on the Al surface. During the first step, the fluoride species of the conversion bath cause a thinning of the natural aluminium oxide film. As a consequence, the electrochemical reduction reaction of the Cr^{6+} to Cr^{3+} and the pH in close proximity of the Al surface is 1.8 to 2.0, which enables the precipitation of chromium hydroxide/oxide. However, the pattern of mud-crack in the immediate vicinity of the particle is different from the coarse cracking seen further away, suggesting that the chromate conversion coating is thinner in this region. Other than the mud-cracks, the chromate conversion coating on the matrix was macroscopically uniform [22].
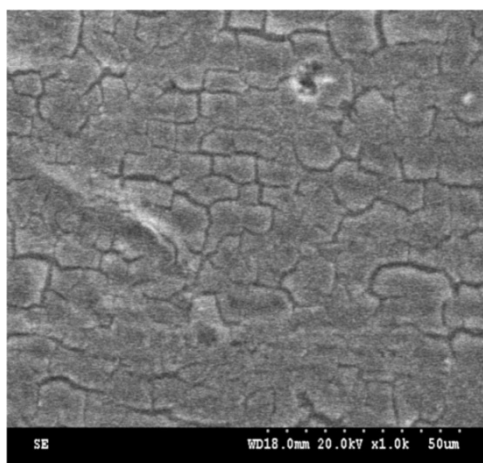

Figure 12. SEM of chromated aluminium surface.

The SEM image of the pickled Al surface is shown in Figure 13. It shows that the surfaces are etched with different sizes of pits and found to be non-uniform.
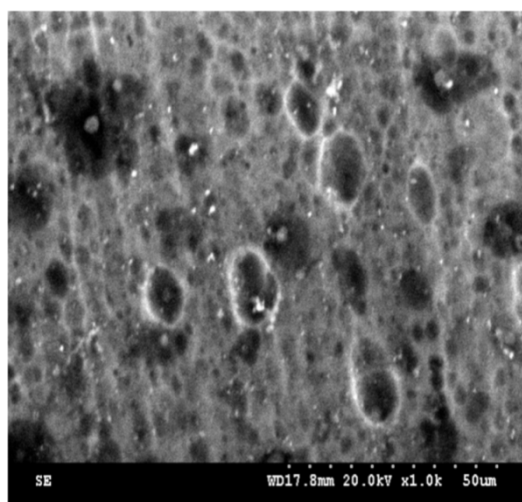

Figure 13. SEM of pickled aluminium surface.

3.4. AFM Studies

The AFM image of the anodized Al surface is shown in Figure 14. It reveals the presence of uniform roughness on the deposited surface[23]. The depth profile occurs between −160 nm and +140 nm, indicating the roughness of the surface. The result is in good agreement with the R_a value of 12.7 nm.
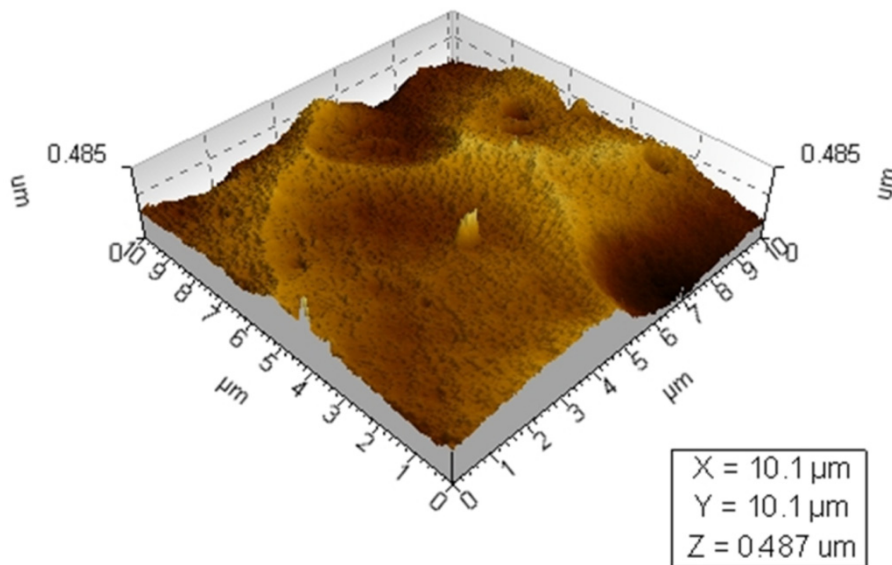

Figure 14. AFM of anodized aluminium surface.

The AFM images on the phosphated Al surface is shown in Figure 15. The R_a value 6.83 nm reflects the smooth nature of the surface.
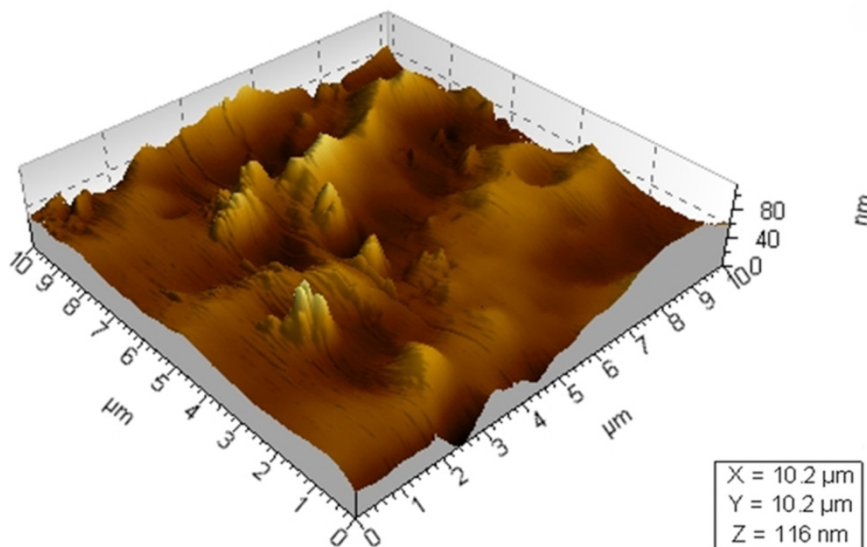

Figure 15. AFM of phosphated aluminium surface.

The AFM image of the chromated Al surface is shown in Figure.16. The R_a value 0.0273, reveals the surface smoothness and uniformity.
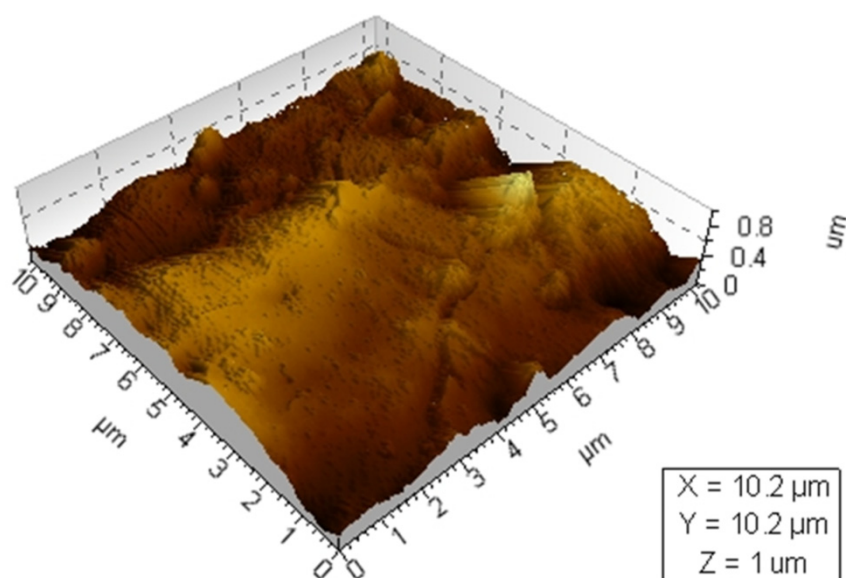

Figure 16. AFM studies on chromated aluminium surface.

The AFM image of the pickled Al surface is shown in Figure 17. The R_a value 19.0 nm clearly indicates that the nature of surface is rough. In general all the pretreated Al surface become smooth after epoxy electrodeposited on Al surface.
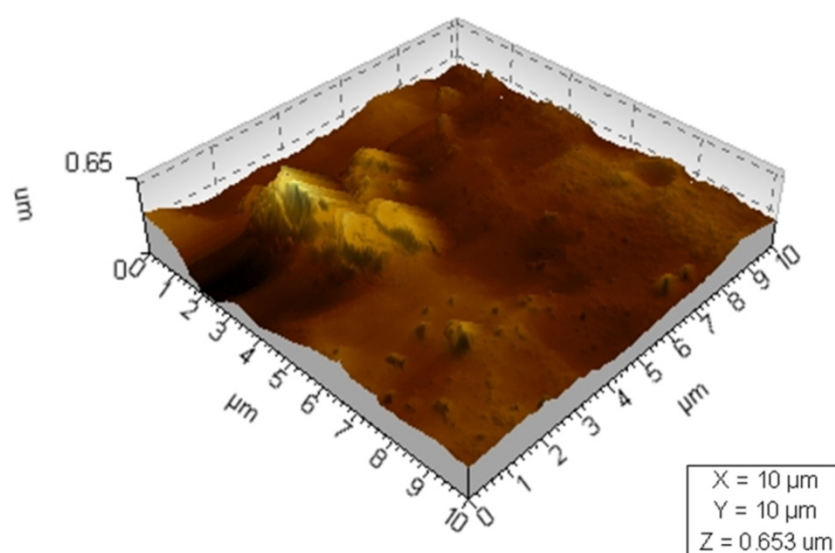

Figure 17. AFM of pickled aluminium surface.

4. Conclusion

We have characterized the epoxy coating and investigated the performance on Al surfaces prertreated by different methods, and the following conclusions were drawn.

EIS studies showed that the cathodic electrodeposition of epoxy coating on anodized Al surface exhibits better corrosion resistance property when compared with the other pretreatments like phosphating, chromating, and pickling.

TGA results indicate that the weight loss is more on the coatings produced at 15 V compared to 25 V, on both the anodized and phosphated Al surface. The reverse trend was observed both on chromated and pickled Al surface.

SEM studies show the porous nature of the anodized surfaces, the spongy and crystalline nature of phosphated, the mud cracks appearance of chromated and the etched surface of pickled Al.

AFM studies revealed that Al surface pretreated by anodized and pickled show the non uniform and roughness, whereas in phosphate and chromate pretreatments show the smoothness and uniform surface.

Anodized and chromated Al surfaces are more suitable for epoxy coatings system for protecting the surface in marine environment.

Acknowledgments

Conflict of Interest

The authors declare that there are no conflicts of interest related to this study.

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