



Morphological analysis and corrosion performance of zirconium based conversion coating on the aluminum alloy 1050



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ABSTRACT

An aluminum alloy 1050 was treated by zirconium-based conversion coatings at different pHs, immersion times, temperatures and Zr concentrations. Corrosion resistance and morphological properties of samples were investigated by potentiostatic polarization, electrochemical impedance spectroscopy (EIS), scanning electron microscope (SEM), energy dispersive X-ray spectrum (EDS) and atomic force microscope (AFM). The results showed that surface treatment of aluminum by zirconium conversion coating caused decrease in corrosion current density and increase in corrosion resistance even at long immersion times. The Zr conversion layer enhanced the adhesion of the epoxy coating to the aluminum alloy surface.

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Introduction

AA1050 aluminum alloy, which represents commercially pure aluminum, offers high corrosion resistance compared to other aluminum alloys. However, it can undergo localized pitting corrosion due to the presence of some alloying elements. Iron and silicon are the major alloying elements presented in the AA1050, which have low solubility in aluminum and form intermetallic particles. The main intermetallics in AA1050 are Al_6Fe , Al_3Fe and $Al_{12}Fe_3Si_2$ which all of them exhibit cathodic activity relative to the aluminum matrix [1–4]. These intermetallics are catalytic sites for cathodic reactions, and at the same time are sites for nucleation of pits. Therefore, the presence of such intermetallics has a detrimental effect on the corrosion resistance [5–7]. AA1050 has been used in conventional engineering applications where the corrosion resistance is required and the mechanical strength is relatively unimportant (e.g., architectural, automotive industries, containers and equipment for food and chemical industries). Therefore, the improvement of AA1050 corrosion resistance has been a topic of great importance.

Conversion coatings are applied on the metals to improve their corrosion resistance and enhance paint adhesion. Chromate and phosphate-containing conversion layers have been commonly used for this purpose [8]. However, they are being increasingly replaced with various alternatives because of several health, environmental, energy, and process disadvantages [9]. Because of these, recently, researches have been focused on development of less toxic alternatives for the chromate-free conversion layers [10–20]. Among different chrome-free coatings developed so far, the conversion coatings based on zirconium and/or titanium have gained acceptance to any extent [11–20]. It was shown that the zirconium oxide/hydroxide deposition on the surface of aluminum alloy occurs preferentially at the cathode sites and the preferential deposition occurs due to the increase of pH at favored locations of cathodic reactions [10–15].

The use of Zr based conversion coating on the aluminum surface influences the adhesion properties of the organic coatings by affecting the surface chemistry and morphology. It causes strong adhesion bonds creation between metal surface and the organic coating [21]. As a result, the interfacial bonds degradation as well as coating resistance reduction occurs with a lower intensity in the presence of Zr coating [19,22].

The present study aims at studying the zirconium-based conversion coatings (ZCC) properties on an aluminum alloy 1050. The anticorrosion properties of the surface treated samples were investigated by a DC polarization technique, electrochemical

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impedance spectroscopy (EIS) and open circuit potential (OCP) measurements. Scanning electron microscopy (SEM), energy dispersive X-ray spectrum (EDS) and atomic force microscope (AFM) were utilized in order to investigate the surface characteristics of samples. The epoxy coating was also applied on the Zr treated substrate in order to evaluate the adhesion properties of the epoxy coating by pull-off test.

Experimental

Materials

Aluminum alloy (1050) samples (2 mm thickness) were supplied by Arak Al Co. The chemical composition of the alloy is given in Table 1.

For surface preparation of aluminum, the chemical materials including acetone, nitric acid (HNO₃) and sodium hydroxide (NaOH) were prepared from Mojallali Co. and Merck Co.

Conversion coating deposition procedure

The surface of aluminum is usually covered by contaminations such as grease, oxide and deformed layers which should be carefully removed from the surface before coating application. Acetone was utilized for chemical degreasing of the aluminum sample. To remove the oxide and any deformed layers, the chemical etching was carried out. The chemical etching was carried out in 5% w/w NaOH solution (alkaline etching) at 50 °C for 3 min followed by washing with distilled water. After that, acid etching (desmutting) was carried out in a 50% v/v solution of nitric acid for 1 min. Finally, samples were subsequently washed with distilled water. All of the prepared samples were kept in a desiccator before coating application.

It has been aimed to apply the ZCC on the surface prepared aluminum samples. In this regard, the aluminum samples were dipped in the hexafluorozirconic acid (H₂ZrF₆) based solution, prepared at different Zr concentrations (C) of 50, 100, 150, and 200 mg/l in deionized water. Using sodium hydroxide (NaOH 0.1 M), the pH of the conversion coating solutions was adjusted to 3.5, 4, 4.5 and 5. The aluminum samples were dipped in the conversion coating bath at different immersion times (t) of 2, 3, 4 and 6 min. The temperatures of treatment bath (T) were 20, 25, 30 and 35 °C. The optimum values of the Zr concentration, pH, immersion time and temperature were obtained after analyzing the samples using different electrochemical and morphological techniques.

Methods

Anticorrosion performance measurement

Potentiostatic polarization technique was utilized in order to investigate the anticorrosion properties of different samples. The test was performed in 3.5% w/w NaCl solution using a potentiostatic model AUTOLAB PGSTAT12. The measurements were carried out in a conventional three electrode cell including Ag/AgCl (3 M KCl) as reference electrode, platinum as auxiliary electrode and aluminum samples as working electrode. The test was carried out on 1 cm² of samples in 3.5% w/w NaCl solution after 30 s immersion. The measurements were done at scan rate and

potential range of 10 mV s⁻¹ and –1300 to –600 mV, respectively. General purpose electrochemical software (GPES) was used to extract corrosion potential (E_{corr}) as well as the corrosion current density (i_{corr}) from Tafel plots.

The corrosion protection properties of the treated samples were also studied by an electrochemical impedance spectroscopy (EIS) model AUTOLAB G1. The test was carried out at amplitude and frequency range of 10 mV and 10 kHz–10 MHz, respectively. A conventional three electrode cell including aluminum specimen as working electrode, platinum as counter electrode and Ag/AgCl (KCl 3 M) as reference electrode was used for this purpose. EIS measurements were carried out at open circuit potential (OCP) on 1 cm² area of each sample after different immersion times in 3.5% w/w NaCl solution. The OCP measurements were also done in NaCl solution at different immersion times in order to investigate the conversion coatings stability against corrosion. To this end, the pretreated samples were exposed to 3.5% w/w NaCl solution up to 15 days immersion. The OCP measurements were done on 1 cm² area using an HIOKI model voltmeter in an electrochemical cell including Ag/AgCl (KCl 3 M) reference electrode and working electrode.

Surface characterization

The morphology and chemical composition of the AA1050, before and after surface preparation and surface treatment, were investigated using SEM (AIS2100) equipped with EDS (SAMx). Moreover, Ambios model AFM (tapping mode) was utilized to investigate the surface morphology and topography of the ZCC.

Pull-off adhesion test

Pull off adhesion tester (model DEFELSKO) was utilized in order to evaluate the adhesion strength values of the epoxy coating applied on different substrates (ASTM D 4541). The measurements were conducted before (dry pull-off strength) and after (recovery pull-off strength) 30 days immersion in the 3.5% w/w NaCl solution. To measure adhesion strength values, the aluminum dollies were glued on the surface of the epoxy coating using two-part Araldite 2015 (Huntsman Advanced Materials, Germany) adhesive. Samples were then kept at ambient temperature for 24 h to insure that the glue fully cured. Finally, a slot was made around dollies and they were pulled at a speed of 10 mm/min normal to the coating surface until the epoxy coating was detached from the aluminum substrate. All tests were carried out using three replicates to ensure the measurements repeatability.

Results and discussion

Open circuit potential measurements during surface treatment

The OCP was measured in the ZCC bath at different pHs and immersion times in order to evaluate the appropriate pH and immersion time of Zr layer deposition on the surface of aluminum. The bath temperature and the Zr concentration during OCP measurement were 25 °C and 100 mg/l, respectively. The results obtained are shown in Fig. 1.

From Fig. 1, three typical regions (I, II and III) can be seen in OCP diagram at different pHs in the short (I), medium (II) and long immersion times (III). At short immersion times (ranged from 0 to 2 min), the OCP declined sharply which can be presumably attributed to the surface activation as a result of hydrated oxide layer dissolution when exposed to the fluorides presented in the ZCC bath. The activation process can be defined by Eq. (1) [12,18,23–25].

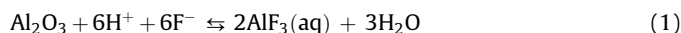


Table 1
Chemical composition of AA1050 (% w/w).

| Al | Si | Fe | Cu | Mn | Mg | Zn | others |
|-------|------|------|------|------|------|------|--------|
| 99.58 | 0.08 | 0.23 | 0.01 | 0.03 | 0.03 | 0.01 | 0.03 |

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