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Effects of different surface cleaning procedures on the superficial morphology and the adhesive strength of epoxy coating on aluminium alloy 1050

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ABSTRACT

This study reports the effects of different cleaning procedures on the surface characteristics of the aluminium alloy 1050 substrates and on the adhesive strength of the epoxy coating to this alloy's surface. The cleaning procedures used in this study were (1) degreasing by acetone, (2) alkaline etching by 5 w/w% NaOH solution and (3) alkaline etching by 5 w/w% NaOH solution followed by acid cleaning by 50 v/v% HNO_3 solution. The surface morphology, chemical composition and topography of the cleaned substrates were investigated by field-emission scanning electron microscope (FE-SEM), energy dispersive spectroscopy (EDS) and atomic force microscope (AFM), respectively. The effectiveness of the cleaning procedures was also studied by polarization test and open circuit potential (OCP) measurements. The surface free energy and work of adhesion were obtained on the cleaned samples using contact angle measuring device. Pull-off test was conducted to evaluate the adhesion strength of the epoxy coating on the aluminium substrates. Results revealed that the surface cleaning of aluminium alloy by alkaline etching followed by acid cleaning method was the most efficient procedure for removing the oxide layer from the surface of aluminium compared to other cleaning procedures. The surface roughness, surface free energy, electrochemical activity and adhesion strength of the epoxy coating to the aluminium surface were significantly increased using this surface cleaning procedure.

1. Introduction

Aluminium has many properties such as low density (2.7 g/cm^3), good conductivity (thermal and electrical) and relatively good corrosion resistance that make it desirable for application in different industries i.e. automotive, aerospace and marine [1]. The corrosion resistance of the aluminium is due to the presence of amorphous oxide layer that naturally forms on the aluminium surface upon exposure to atmosphere. This layer is responsible for the metal surface passivation. However, the oxide layer has a limited thickness and flaws exist in the natural oxide film, which can act as sites for the film breakdown. Moreover, this layer is not stable in acidic ($pH < 4$) or alkaline ($pH > 9$) environments as well [2,3]. Alloying is a process to improve the mechanical strength of the aluminium. Alloying elements and other impurities partially go to the matrix and partly form intermetallic phases [1]. The alloying process of aluminium may increase its mechanical strength, making it useful for a wider applications, but the corrosion resistance of the alloy is often much lower than the pure aluminium. It has been shown that the oxide layer formed on the intermetallic particles may be thinner or non-existent, which leaves these areas more prone to attack [4]. Moreover, some intermetallic particles were found to be nobler than the matrix and enhanced the development of pitting. These particles are nucleation and growth sites for pitting [5–8].

In addition to oxide layer, the surface of the aluminium contains a deformed layer which arises from thermomechanical process. This layer is non-uniform in thickness and composition, and also includes various cracks and voids. This deformed surface layer has different electrochemical properties and microstructural characteristics compared with the underlying bulk microstructure that affects its corrosion resistance susceptibility. This layer has high electrochemical reactivity, in particular and a strong susceptibility to localized corrosion [9]. The chemical composition of the aluminium substrate i.e. the type and concentration of the intermetallics can affect its corrosion resistance [6].

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AA1050 aluminium alloy, which represents the commercially pure aluminium, has been used in the conventional engineering applications where the corrosion resistance is required and the mechanical strength is relatively unimportant (e.g., architectural, automotive industries and containers and equipment for the food and chemical industries). However, it can undergo localized pitting corrosion due to the presence of some alloying elements such as iron (Fe) and silicon (Si) [10,11]. Therefore, the improvement of the AA1050 corrosion resistance has been a topic of great importance.

Matter and Kozhukharov [12] have studied the effect of the preliminary treatment on the properties of the oxide film and its influence on the corrosion behaviour of the AA2024 in corrosive media. They found that the preliminary treatment had significant influence on the features and other behaviours of the alloy in corrosive medium.

Using organic coatings on the surface of aluminium substrate is a common way to minimize its corrosion susceptibility. However, the effectiveness of the corrosion protection properties of the coating may be influenced by the surface properties of the aluminium. The poor adhesion of the organic paints on the surface of the aluminium has been become one of the most important challenges of the researches in last decades. This is because of the naturally formed surface oxide as well as deformed layer on the surface of the aluminium. Surface preparing is one of the most important ways governing the adhesive strength of the organic coatings bonded to the aluminium alloys through removing surface oxide and deformed layer obtaining fresh, clean and reactive aluminium surface. This can provide the maximum bonding capacity towards the subsequent treatments [6,13–17]. Prolong and Urena [16] have examined several etching procedures for the improvement of adhesive bonding of aluminium alloys. They found that the etching treatment affects thickness, composition, aspect and porosity of oxide layer formed, which also depends on the alloy nature. Their results indicated that the effectiveness of the applied pre-treatment not only depends on the aluminium surface nature but also on the adhesive nature.

In the present paper, different cleaning procedures are utilized to clean the AA1050 surface. The degree of aluminium surface cleaning was characterized by field-emission scanning electron microscope (FE-SEM) equipped with energy dispersive spectroscopy (EDS), atomic force microscope (AFM), potentiostatic polarization and open circuit potential (OCP) measurements. The surface free energy and work of adhesion were also obtained by measuring water contact angle on the cleaned samples. Additionally, the effect of different cleaning procedures on the adhesion strength of the epoxy coating on the aluminium alloy was also studied by pull-off test.

2. Experimental

2.1. Materials

The aluminium alloy 1050 sheet (2 mm in thickness) used in the present study was purchased from Arak Al Co. The composition of the alloy is given in Table 1.

The chemical cleaning baths were prepared using acetone, nitric acid (HNO_3) and sodium hydroxide (NaOH) supplied by Mojallali Co. and Merck Co.

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

2.2. Sample preparation

2.2.1. Surface cleaning procedures

The metal plates underwent three cleaning procedures including degreasing by acetone (S1), alkaline etching by 5 w/w% NaOH solution for 3 min at 50 °C and then washing with distilled water and alkaline etching by 5 w/w% NaOH solution for 3 min at 50 °C followed by acid cleaning by 50 v/v% HNO_3 solution for 1 min at ambient temperature and finally washing with distilled water (S3).

2.2.2. Epoxy coating application

The adhesion of the epoxy coating to the differently cleaned aluminium substrates was studied. The epoxy resin used was based on bisphenol-A (Araldite GZ7 7071X75) in a xylene solution. The solid content, epoxy value and density of the resin were 74–76%, 0.1492–0.1666 Eq/100 g, and 1.08 g/cm^3, respectively. To prepare the epoxy coating, the epoxy resin was mixed with a polyamide hardener (in which the ratio of the epoxy resin to hardener was 70:40 w/w). Additives like leveling agent (BYK-306) and defoamer (Efka-2025) were added to the coating formulation to enhance the coating film formation performance. Finally, the coatings prepared were applied on the aluminium samples by film applicator. Samples were then cured in an oven set upped at 120 °C for 30 min. A dry film thickness of 40 ± 5 μm was measured on the cured samples by a digital coating thickness gauge (Elcometer 456).

2.3. Characterization and testing

2.3.1. Surface characterization

The surface morphology and chemical composition of differently cleaned substrates were investigated using FE-SEM (Mira) equipped with EDS (SAMx). Moreover, Ambios model AFM (tapping mode) was utilized to investigate the surface topography of the samples. Static contact angles were measured on different samples by an OCA 15 plus type contact angle measuring device using distilled water as the probe liquid at temperature and humidity of 25 ± 2 °C and 30 ± 5%, respectively. In this regard, a small drop of distilled water (2–3 μl) was applied on the surface of samples. The shape of droplet was recorded by a Canon type digital camera after 10 s. The images were transmitted to a personal computer for evaluation. Using an image analysis system (G2/G40), the contact angle values were calculated.

2.3.2. Electrochemical measurement

Potentiostatic polarization technique was also utilized in order to investigate the anticorrosion properties of the samples. The test was performed in the 3.5 w/w% NaCl solution using a potentiostat model AUTOLAB PGSTAT12. The measurements were carried out in a conventional three electrode cell including Ag/AgCl (3M KCl) as reference electrode, platinum as auxiliary electrode and aluminium samples as working electrode. The test was carried out on 1 cm^2 of the samples in the 3.5 w/w% NaCl solution after 30 s immersion. The measurements were done at scan rate of 10 mV s^{-1}. To ensure the repeatability of the measurements, the test was done on three replicates.

The OCP measurements were done in the 3.5 w/w% NaCl solution at different immersion times on the differently cleaned samples. 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 aluminium sample.

2.3.3. Pull-off adhesion test

PosiTest pull-off adhesion tester (DEFELSKO) was utilized to evaluate the adhesion strength values of the epoxy coating applied on differently cleaned substrates (ASTM D 4541). For this purpose,