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Effect of Heat Treatment and Sulfuric Acid Anodization on Corrosion Resistance of Aluminum Alloy (AA7075)

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Abstract

This paper, studied the effect of heat treatment and anodization on corrosion resistance of aluminum alloy 7075 (AA7075), with a view to improving its corrosion resistance. Microstructure and micro hardness of the anodic film of the samples were studied with the aid of optical metallurgical microscope and automated micro hardness testing machine. Linear polarization methods were used to assess the corrosion behaviour of the alloy in 0.5M HCl. The microstructure of the annealed sample showed formation of dendrites while precipitation hardened samples in palm kernel oil and SAE 40 engine oil showed precipitates of $MgZn_2$. The SEMS result showed pores and micro cracks on the surfaces of the anodized samples, with the as cast and anodized sample in sulfuric acid exhibiting most compact with few pores. The as cast and sulfuric acid anodized sample shows highest micro hardness value of 205.33 HV, while the least value of 150.67 HV was recorded in sample precipitation hardened in SAE 40 engine oil and anodized in sulfuric acid. Analysis of the potentiodynamic polarization data and curves showed a linear relationship (decrease in icorr, decreases the corrosion rate) between current density and the corrosion rate in all the samples. Higher polarization resistance of 15.093 Ω/cm_2 was recorded by the as cast and Sulfuric acid (SA) anodized sample while the precipitation treated in SAE 40 engine oil plus SA anodized sample recorded lowest polarization resistance of 5.2311 Ω/cm_2. Heat treatment alone improves corrosion resistance of AA 7075 in 0.5 M HCl solution but heat treatment plus SA anodization does not improve corrosion resistance in the same environment.

Keywords: heat treatment, anodization, micro hardness, corrosion, AA7075

1. INTRODUCTION

Metals constitutes eighty percent of element in the periodic table and react with oxygen to form basic oxides, it is significant among other engineering materials such as composite, polymer and plastic due to their physical, mechanical and chemical properties, suitable for wide range of applications. Aluminum is the second most widely used metal, highly valuable due to its unique combination of properties such as low density (approximately 2.7 kg/cm^3), high ductility, and improve mechanical strength achieved by suitable alloying and heat treatments [1, 2].

Aluminum is broadly used in the automotive, air-space, building, and electronics industries and environmentally beneficial due to weight reduction [1]. However, when exposes to the atmosphere, its readily oxidized to produce a thin and fragile oxide layer which tends to protect the metallic surface (Al_2O_3) and prevent further oxidation process.

Historically, steels and alloy have been predominantly used in building and automobile industries since 1920's. Recently, aluminum alloy has been decently used in engineering structural applications owing to its wide range of characteristics such as light weight, non-rusting properties, reasonably good strength and easy fabrication, modern metallurgical control of structure and properties [3].

Although, metals are very important engineering materials, but the serious setback to their mechanical properties is corrosion. The effect of unchecked corrosion therefore is not limited to the state of corroding utility itself but also influences

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economic, health, safety, technological, and cultural consequences to our society [4, 5]. The basic cause of corrosion is the instability of metals in their refined forms. The metals tend to revert to their natural states through the processes of corrosion. One of the most important factors influencing corrosion is the difference in electrical potential of dissimilar metals when coupled together and immersed in an electrolyte. This potential is due to the chemical natures of the anodic and cathodic regions.

In addition, agitation acts to increase the corrosion rate by bringing fresh corroding solution into contact with the metal. Also, differences in potential from point to point on a single metal surface cause corrosion known as local action and may be due to impurities on the surface or differences in the surface structure or environment. Other factors such as the presence of other ions in solution, the temperature of the solution and the existence of stray electric current- may materially affect corrosion rate [6, 7]. As is true of the various forms of corrosion, there are many different methods of corrosion prevention and control.

A proven chemical surface engineering technology to increase corrosion and wear resistance is anodizing. This is a chemical surface engineering technique that oxidizes and converts the surface of aluminum to a uniform and continuous alumina oxide film [8]. Alumina has a relatively high hardness (2.9 – 5.9 kg/mm^2) and is chemically inert [9–12].

Anodization changes the microscopic texture of the surface and changes the crystal structure of the metal near the surface. Anodic films are generally much stronger and more adherent than most types of paint and metal plating, but also more brittle. This makes them less likely to crack and peel from aging and wear, but more susceptible to cracking from thermal stress.

The amount of Al_2O_3 formed during anodization is directly proportional to the current density and time while the growth of the film depends on the chemical composition. Concentration of the anodizing electrolytes has little or no solvent action on the oxide coating so that very thinning films usually known as barrier layer type coating are formed, the thickness of which is solely dependent on applied voltage. These types of coatings are typically produced in solutions of borate, boric acid or titrate. Sulfuric acid is an example of electrolytes that has solvent action on the formed coating. Porous film is also formed as the oxidation process continues leading to the formation of relatively thick film [6, 13]. The aim of this work is to investigate the effect of heat treatment and sulfuric acid anodization on corrosion behavior of aluminum alloy 7075 (AA 7075) with a view to improving its corrosion resistance for engineering applications.

2. MATERIALS AND METHODS

2.1. Materials

The aluminum alloy 7075 was cast using stir casting method and the result of the elemental composition is presented in Table 1.

The corrosive medium used is 0.5M HCl solution. Other materials include commercial aluminum scrap (260 g) and copper cut-out (8 g) sourced from NOCACO Aluminum company Kaduna, Zinc scrap (16 g), Magnesium scrap (5 g), abrasive emery grinding paper, ACS Reagent Grade Ethanol, distilled water, Analytical reagent grade Sulfuric acid (H_2SO_4) solution (95% concentration, 1.83 g/ml), Analytical Reagent grade (90% concentration, 1.48 g/ml) Nitric acid (HNO_3) solution, Laboratory Reagent grade, ≥ 9 8% Sodium hydroxide (NaOH), Analytical Reagent grade, ≥ 37% Hydrochloric acid (HCl) solution, Laboratory Reagent grade, 99.99% Chromium trioxide (Cr_3O) crystals, SAE 20 W/50 engine oil (AMMASCO), Freshly extracted Palm Kernel oil (4 liters) from Edati in Niger State.

2.2. Equipment

Equipment used for this research work include: Electric furnace for heating: Brand/make/model: search Tech instruments, 5X-5-12 Box-Resistances No. 2392, Charcoal fired crucible furnace, Energy Dispersive X-ray Fluorescence (EDXRF) machine, model: Minpal 4 No. DY 1055, Photographic Visual Metallurgical microscope: Model No. NJF-120A, Scanning Electron Microscope (SEM): Model No. EVO MA- 10, manufacture by Carl, Automated micro hardness tester: Model MV1-PC, Identification No Mh-vA with Max/Min Limit of 220/80 HV, Load 0.3 kgf and Machine No 07/2012-1329, Potentiostat/Galvanostat type PGSTAT 204, serial no. AUT50280 made in Netherlands 75W/60Hz, Lathe machine, 12-volt Battery (Delkor): NS 60 LSMF, AMP. HR (20 HR)- 45, RESERVE CAPACITY (MIN)- 75,12V 45 Ah, N 45, 2 mm diameter electric wires, Digital Multimeter Model 57030: DC voltage range: 200 mV - 600 V, DC current range: 200 μA – 10 A, AC voltage range: 200 V – 600 V, Resistance range: 200 Ω – 2 MΩ, Digital laboratory pH meter (Range: -2.000 to 20.000 pH, accuracy of ± 0.002), Thermometer (Mercury/Red spirit filled), Range: -10 to 110 °C, Sensitive laboratory electric weighing balance Model No. FA2004, Rating load: 200 g, Actual scale dividing values: 0.1 mg, Accuracy: 200 g, 0.1 mg, Voltage: 220 v/50 hz, 2-liter capacity Pyrex beaker, Plastic container, Desiccator.

Table 1: Chemical Composition of Cast AA 7075.

Element	**Composition wt.%**
Zn	5.561
Mg	1.049
Cu	1.06
Fe	1.1
Ca	0.02
Ni	0.0091
Others each	0.0009
Al	91.2

2.3. Methods

The cast rods were machined to 14 mm diameter and 10 mm in length (cylindrical in shape). The cylindrical samples were made smooth by thoroughly grinding on emery abrasive grinding papers. Some of the prepared samples were then subjected to aging by heating in a Box-Resistance Furnace Model No 2392 to solutionized at 510°C ± 5°C, soaked for 3 hours and then 9 samples were quickly removed and plunged into separate containers containing different quenching media (palm kernel oil and SAE 40 engine oil) at ambient temperatures as specified in [14]. Thereafter, the quenched samples were aged at a temperature of 170°C ± 5°Cas prescribed in [14] standards and then allowed to cool in air.

2.4. Condition for Anodization

Prior to anodization, samples were subjected to alkaline cleaning in 5% NaOH solution for 5 minutes followed by desmutting in 25% HNO_3 for 2 minutes, washed, rinsed, dried and weighed. Cleaned and weighed (W1) AA 7075 samples were then anodized in solution of 200g/l H_2SO_4 solution at 12 V for 30 minutes at 30°C [5].

2.5. Experimental Setup for Anodization

The anodizing facility permitted the control of the anodizing parameters such as current, voltage and temperature. The anodizing setup consisted of a transparent plastic container. A 2-litre capacity Pyrex beaker was placed inside a larger transparent plastic container and the space between them was filed with water. The water contained in the transparent plastic container bath increased the cooling effectiveness and acted as a buffer to maintain constant anodizing solution temperature. A mechanical stirrer was used to stir the anodizing solution and ensure a uniform electrolyte temperature throughout. This was effective to dissipate quickly the heat evolved at the specimen electrolyte interface during anodizing.

Both anode (AA 7075) and cathode (stainless steel 0.50 mm thick with dimension of 120 mm length by 110 mm breath) were connected with electric wires and partially dipped into the acid electrolyte solution (Sulfuric acid), which act as a medium for transporting the ionic species. Both anode and cathode were then connected to the positive and negative terminals of a 12 V battery which act as a D.C power supply. A multimeter was connected in series with the electrode to measure the current and voltage passing through [15]. When the voltage was applied (for 30 minutes) between the anode and cathode, pores were nucleated and started to grow on the aluminum 7075 alloy surface.

2.6. Determination of coating thickness

After anodization, specimen with the anodic coating film were weighed (W_2) and then subjected to stripping in a solution containing 20 g/l of chromium trioxide and 35 ml/l of phosphoric acid, for 30 minutes at 100°C, rinsed, dried and weighed (W_2). From these weights the coating ratio were calculated as follow:

Coating ratio,

$$C_R = \frac{W_2 - W_3}{W_1 - W_3} \tag{1}$$

Where $W_2 - W_3$ is weight of the anodic oxide coating and $W_1 - W_3$ is the total weight of Al 7075 reacting.

The thickness of the anodic coating was calculated from the weight of anodic coating, surface area and density of the anodic film as follows:

$$T = \frac{W_1 - W_3 \times 10^4}{Ax\rho} \tag{2}$$

Where A is the surface area of the specimen (cm^2)

$$A = 2\Pi Rh + 2\Pi(R_2 - r_2) + 2\Pi rh \tag{3}$$

ρ is the density (g/cm^3) which is about 2.6 g/cm^3, T is the coating thickness of anodic film (μm), R is the radius of the coupon sample, r is the radius of the drilled hole on the coupon sample and h is the height of the specimen. This is in accordance with the method of [16].

2.7. Determination of Micro Hardness of the Anodic Film

The Vicker micro hardness test was performed according to [17] by applying the loads of 10 gf, 25 gf and 40 gf at 15gf interval during 10 seconds.

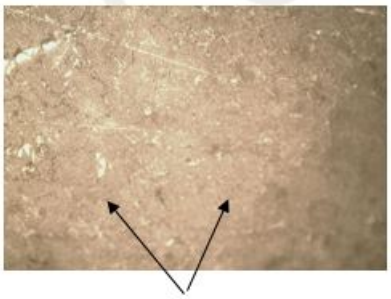
2.8. Potentiodynamic Polarization Analysis

The corrosion behavior of the anodized samples formed in various anodizing conditions were evaluated through Potentiodynamic polarization method in which corrosion parameters such as corrosion potential (ECorr), Corrosion current (ICorr), Polarization resistance (Rp) and corrosion rate (CR) for the AA 7075 samples after their immersion in hydrochloric acid (HCl) test solution for 30 minutes. A potentiostat was coupled to a computer, a glass corrosion kit with graphite rods as counter electrode and a saturated Ag/Ag 3M Kcl electrode was also used as the reference electrode (SCE). The 14 mm × 10 mm test samples (working electrodes) were tightly mounted and positioned at the glass corrosion cell kit leaving the average of 0.8262 cm^2 surface in contact with the electrolyte. The process was carried out by applying current density of 40 A/cm^2, scan rate of 0.001, start and end potential of -1.500 and 1.500 respectively. Polarization curves were plotted using Auto lab data acquisition system, both the corrosion rate and potential were estimated by Tafel extrapolation method using both the anodic and cathodic branches of polarization curves according to [18, 19].

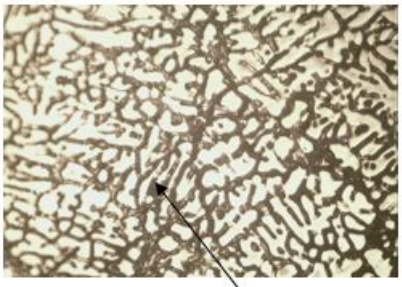
3. RESULTS AND DISCUSSION

3.1. Microstructure Analysis

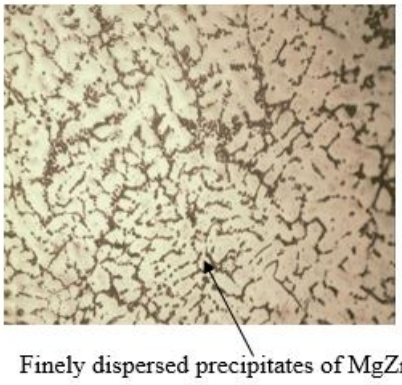
The micrograph of the as cast, heat treated and anodized AA7075 specimen viewed under metallurgical microscope and the scanning electron microscope (SEM) are presented in Fig. 1a – 1d and Fig. 2a – 2d respectively.



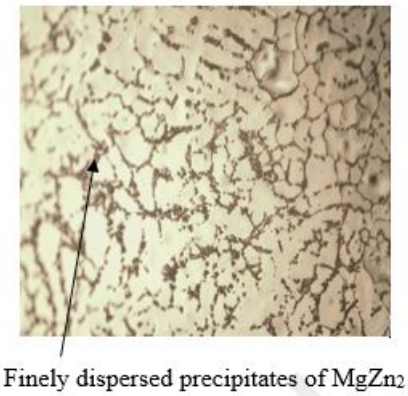
(a) cast



(b) annealed



(c) Precipitation Treated in palm kernel oil



(d) Precipitation hardened in SAE 40 engine oil

Figure 1: Optical micrograph of AA 7075, showing α-Al (white) and intermetallic phase (black). Mag X100: (a) as cast, (b) annealed, (c) Precipitation Treated in palm kernel oil, (d) Precipitation hardened in SAE 40 engine oil.

4. DISCUSSION

4.1. Chemical Composition Analysis

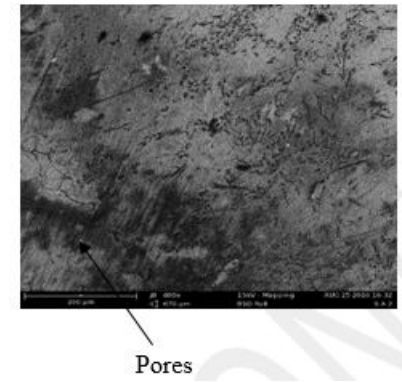
The chemical composition analysis of the cast AA 7075 was carried out using x-ray fluorescence (XRF) analysis. The result presented in Table 1 shows slight difference in compositions of some elements as quoted in [20]. These differences may be due to impurities and variation in melting temperatures of the charged materials.

4.2. Effect of Heat Treatment on Microstructure

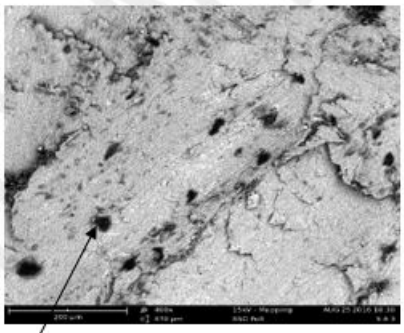
The micrographs of AA 7075 specimen are presented in Fig. 1a – 1d, for as-cast, annealed and precipitation hardened conditions. The microstructure of this type of alloys is generally said to be characterized by η precipitates (nominally $MgZn_2$) [21, 22] as can be observed in Fig. 2a – 2d. The microstructure of the as cast sample (Plate 1a) shows micro segregation of $MgZn_2$ in aluminum matrix which corresponds with the result of [23]. From the micrographs, it can be seen that the microstructures of the precipitation treated alloys (Fig. 1c and 1d) have finer grains and consequently more grain boundaries than the as-cast and annealed alloys. This is based on the fact that there is an increase in the volume of the precipitated intermetallic compounds during precipitation hardening process and also refinement of the precipitated constituent particles [24]. The grains of the palm kernel oil quenched sample are finer when compared with the grains of SAE 40 engine oil quenched sample, this is due to slower cooling rate of the palm kernel oil. The presence of dispersed precipitate of $MgZn_2$ correspond with the result of [25–27] who discovered that aging heat treatment of Al-Zn-Mg-Cu alloy lead to the formation $MgZn_2$ intermetallic phase in the structure. In their study on Investigation of the Quenching Properties of Selected Media on 6061 Aluminum Alloy, [28] reported that increase solution treatment temperature was found in improved soluble precipitates in the alloy, and the heat extraction capacity of the quenching medium also contributed to the formation of fine precipitates. The elimination of micro segregations after annealing and precipitation treatment operations in the present investigation is in agreement with the findings of [29].

4.3. Effect of Heat Treatment on the Anodic Film Formation

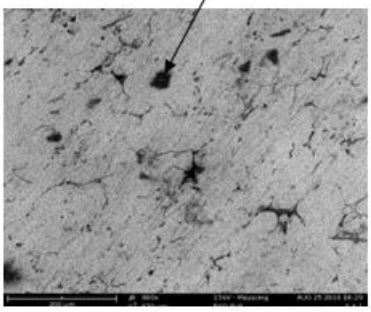
In this study, the effect of surface microstructure resulting from different heat treatment processes on the progress of anodic film on AA 7075 was analyzed. Figures 2a – 2c showed the SEM micrographs of the surface morphologies of sulfuric acid (SA) anodized AA 7075 (subjected to different heat treatment processes). The surface of the as cast sample exhibited the most compact layer with fewer surface pores [30]. Micro cracks were seen on the surface of the SA anodized specimen. These micro cracks on the surfaces of the anodized layers may likely be caused by internal



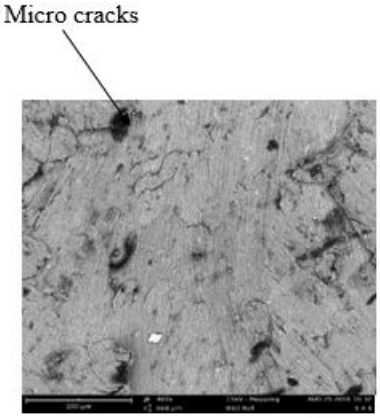
(a)



(b)



(c)



(d)

Figure 2: SEM micrograph of as cast + SA anodized (a), Annealed + SA anodized sample (b), Precipitation treated sample in palm kernel oil + SA anodized (c) and Precipitation treated sample in SAE 40 engine oil + SA anodized sample (d).

stress generated by the growth of the oxide at the substrate/oxide interface [31]. This result is in agreement with the observation of [32].

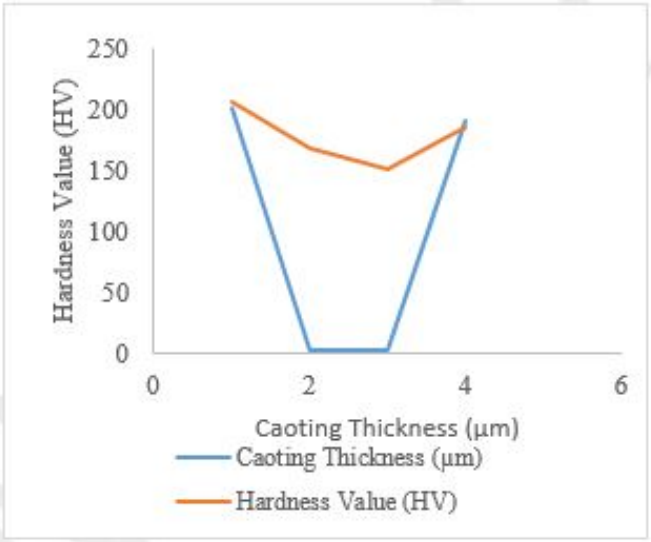


Figure 3: Variation of micro hardness and coating thickness of the anodic film formed on the heat treated and sulfuric acid anodized AA 7075 samples.

4.3.1. Micro hardness and coating thickness of the anodic film

Figure 3 shows variation of hardness value with the coating thickness of the anodic film formed on the as cast, heat treated and heated and anodized samples in sulfuric acid (SA). Decrease in hardness with increase in coating thickness was observed. Highest micro hardness value was recorded at 186 HV for annealed plus SA anodized sample. At very high coating thickness (248.38 μm), the micro hardness is maximum and hence the rate of formation of coating is more. The difference in weight gain between various coatings correlates their surface morphology [33]. This result is in agreement with the result put forward by [34].

4.4. Effect of Heat Treatment and Anodization on the Corrosion Resistance

Figure 4 show Potentiodynamic polarization curves of the heat treated and anodized AA 7075 samples exposed to 0.5 M HCl solution. The corrosion rate of all the samples studied showed prolong active region (anodic).

Observations from the results indicated that the corrosion rates are very similar in trend for all the examined samples irrespective of the treatment. This is because the samples were from the same alloy which was cast and solidified in similar conditions of cooling rate. The corrosion current densities (jcorr) were obtained from polarization curves in Fig. 4 by Tafel plots using both cathodic and anodic branches of the polarization curves. The corrosive environment demonstrated a decreased in corrosion rate and current density while the heat treatment processes increases –E (V) and R_P (Ω cm^2) of the samples. This result agrees with the result of [35, 36]. The heat treated

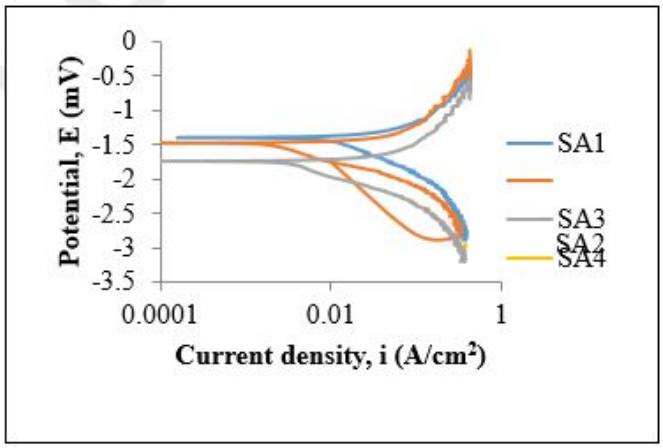


Figure 4: Potentiodynamic polarization curves of heat treated and sulfuric acid anodized AA7075 samples exposed to 0.5M HCl solution.

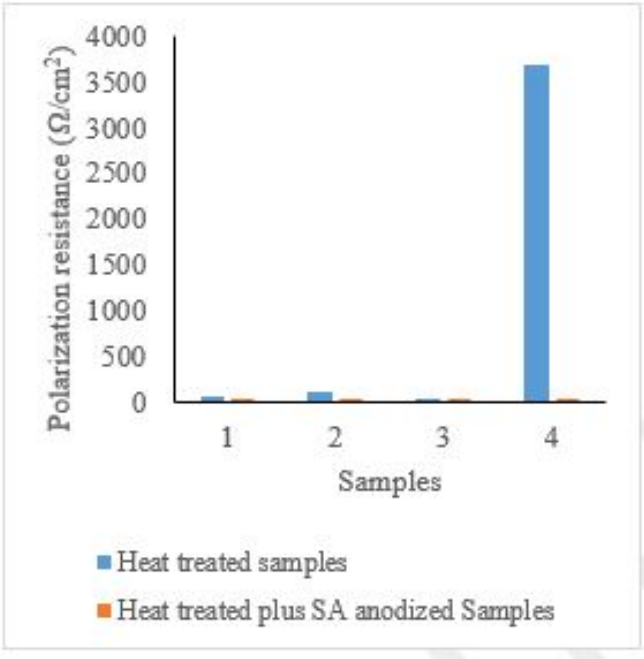


Figure 5: Variation of the effect of heat treatment on polarization resistances of AA 7075 before and after anodization in sulfuric acid.

AA 7075 samples in test environment showed that corrosion rate decreases from 32.388 mm/yr to 4.8893 mm/yr for sample annealed sample plus sulfuric acid (SA) anodization to SAE 40 engine oil precipitation treated plus SA anodization respectively.

Highest potential (-1976 mV) was observed for the as cast plus sulfuric acid anodized sample while the highest polarization resistance (15.093 Ω/cm^2) was observed for the as cast plus SA anodized sample. This result is in agreement with the result of [37].

Lowest polarization resistance (5.2311 Ω/cm^2) was recorded by the sample precipitation treated in SAE 40 engine oil plus SA anodized sample as can be seen in Fig. 5.

Samples subjected to heat treatment and SA anodization recorded lower corrosion resistance when compared with the samples subjected to heat treatment alone (as can be seen in Fig. 5), which suggest that heat treatment plus SA anodization does not improve the corrosion resistance of AA 7075. Higher polarization resistance (15.093 Ω cm^2) recorded by the sample as cast and SA anodized which may be attributed to few pores on its surface as can be seen in Fig. 2a. It was found that the anodizing potential is critical in determining the anticorrosion performance; lower potential generates finer pores that provide enhanced corrosion protection compared with the larger pores generated at higher potentials [38].

5. CONCLUSIONS

The results obtained from this research indicates significant role of heat treatment, heat treatment and anodization on the corrosion behavior of AA 7075. The corrosive medium did not penetrate the anodic film uniformly but preferentially at local sites, resulting in localized corrosion of the anodized alloy. Based on the results presented, the following conclusions are made:

1. The chemical composition analysis of AA 7075 that was carried out (Table 1) shows the presence of 91.2% Al, 5.561% Zn, 1.049% Mg, 1.06% Cu, 1.10% Fe, 0.02% Ca and 0.0091% Ni, and 0.0009% other each with slight difference in the compositions of some elements quoted in [20].

2. Heat treatment leads to the formation of $MgZn_2$ phase on AA 7075. Annealed sample gives rise to non-uniformly distributed and dissolved coarse grains of $MgZn_2$ phase. However, the precipitation treatment in palm kernel oil and SAE 40 engine oil samples showed finely dispersed precipitates of $MgZn_2$ in the aluminum matrix. The as cast sample shows Zn-Al-Cu intermetallic phases.

3. The annealing plus SA anodization only improves the coating thickness of anodic film formed on AA 7075. Also, the as cast plus anodized in SA samples appeared to have better micro hardness; this suggest that heat treatment does not improve coating thickness and micro hardness of AA 7075.

4. The SEM micrograph of the samples shows pores and micro cracks irrespective of the heat treatment the samples were subjected to.

5. Compared with the as cast, we can say that heat treatment alone improves polarization resistance of AA 7075 in 0.5 M HCl solution but heat treated plus SA anodization does not improve polarization resistance in the same environment.

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