ANODIZING OF ALUMINUM-MAGNESIUM ALLOY 5052

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ABSTRACT

Aluminum-magnesium alloy type 5052 has been anodized using sulfuric acid as an electrolyte. The effect of current density in the range of 2-3 Amp/dm^2, electrolyte concentration in the range of 2-6 Normality, electrolyte temperature in the range of 15-25 °C and time of exposure in the range of 20-60 minutes on the thickness of the anodic film are studied. The experimental data was fitted in terms of the oxide film thickness and the coefficients of second order polynomial are estimated. Optimum conditions of the studied variables are predicted and found equal to 3 Amp/dm^2, 2 Normality, 15 °C and 60 minutes. The anodized specimens are sealed in two different mediums' i.e. in hot distilled water that gave transparent coatings and in potassium dichromate that gave colored coatings. Specimens at optimum conditions were produced and their corrosion resistance in CASS (copper-accelerated acetic acid salt-spray), oxidizer and fuel solutions was measured. Also their hardness and surface roughness were measured. Furthermore, comparison study between the anodized specimens subjected to the two sealing mediums and the untreated specimens was carried out.

Current density and time of exposure has shown positive dependence of greater importance in comparison with the other two variables (i.e. concentration and temperature of electrolyte). Besides, the corrosion rate for the uncolored specimen was found less than for the colored and untreated specimens. In contrary, the hardness value for the colored specimen was found greater than for the uncolored and untreated specimens respectively.

INTRODUCTION

Anodizing differs from electroplating in two significant respects. In electroplating, the work is made the cathode, and the metallic coating is deposited on the work. Whereas, in anodizing, the work is made the anode, and its surface is converted to a form of its oxide that is integral with the metal substrate (Frederick, 1978). Therefore, anodizing differs from electroplating where a layer of metal is applied over the basis metal surface. Anodic oxidation, or anodizing, is an electrolytic process for producing very much thicker oxide coating whose improved physical and chemical properties have greatly increased the field of application for aluminum. In this process the specimen is made to become the anode in an electrolyte, which may be chromic acid, oxalic acid, or sulfuric acid with a non-dissolved metal as a cathode. When the anode is aluminum, the cathode, in commercial practice, is either aluminum or lead. When current is passed through the electrolyte, such as H2SO4, most of the oxygen that would have been liberated combines with the aluminum to form a layer of porous aluminum oxide while hydrogen is liberated at the cathode. The amount of aluminum oxide formed is directly proportional to the current density and time, i.e. to the quantity of electric current used. The progress of the formation of the anodic coating depends upon the chemical composition of the anodizing electrolyte and the chosen conditions of electrolysis (Henely, 1982).

The anodized layer is built up from the base of the film but the acids used for the anodic oxidation processes have a solvent action on the aluminum oxide film so that a porous cell structure is formed. Under normal conditions about half the aluminum converted to aluminum oxide is dissolved by the anodizing solution (Norton, 1998). High current densities give thicker film, but when a certain film thickness is reached, the precise thickness depending on the alloy being anodized (i.e. the rate of film formation balances the rate of dissolution and the film will not grow any thicker). However, the overall dimension of the component will be reduced. The thickness of the barrier layer and the cell wall are proportional to the voltage applied.

The size of the pore or cell is dependent on three main inter-related factors: solution concentration, solution temperature and applied current density. By varying these factors anodized film can be produced for different purposes (Darby, 1983). In this investigation the thickness of anodic film of aluminum-magnesium alloy (5052) in sulfuric acid anodizing process was studied in order to study the effect of film formation on corrosion resistance of the alloy against different environments. The effect of sealing step on the anodized specimens by hot water and by potassium dichromate would also investigated.

EXPERIMENTAL WORK

The proper technique for planning a system of more than three variables is "Central Composite Rotatable Design". The total number of treatment combinations is equal to (2k + 2k + 1), where k is the number of variables, plus additional further treatments to take the lack of fit and experimental error into account. These designs consists of a 2k fractional (i.e. coded to the usual ± 1 notation) augmented by 2k axial points, i.e. (±α, 0, 0, …, 0), (0, ±α, 0, …, 0), (0, 0, ±α, …, 0), ….., (0, 0, …, ±α,) and center points (0, 0, 0, …, 0).

A preliminary step is to set up the relationships between the coded levels and the corresponding real variables. These relationships are as follows (Box and George, 1978): -

$$X_{\text{coded}} = \frac{\left[X_{\text{actual}} - X_{\text{center}}\right]}{\left[\dfrac{X_{\text{center}} - X_{\min}}{\sqrt{k}}\right]} \qquad (1)$$

The experimental work was designed in the following experimental ranges:

1. Current density (DC) ranged from 2 to 3 Amp/dm2.
2. Concentration of the electrolytic solution (H2SO4) ranged from 2 to 6 N.
3. Operating temperature ranged from 15 to 25 oC.
4. Contact time ranged from 20 to 60 min.

The central composite rotatable design of four variables was used. The coded levels were related to real process values of these variables as follows:

$$X_1 = \frac{A - 1.25}{0.125} \qquad (2)$$

$$X_2 = \frac{C - 4}{1} \qquad (3)$$

$$X_3 = \frac{T - 20}{2.5} \qquad (4)$$

$$X_4 = \frac{t - 40}{10} \qquad (5)$$

Where: A is current density (DC) in Amp/dm2, C is the concentration of electrolytic (H2SO4) solution in Normality (equi./liter), T is the operating temperature in oC and t is time of contact in min.

The working ranges of coded and corresponding real variables are listed in Table (1). Thirty-one experiments were carried out in a sequence shown in Table (2) where the coded values +2, -2, 0 present the maximum, minimum and average values respectively.

Table (1) Working range of coded and corresponding real variables

Coded Level	Current Density (Amp/0.5 dm^2)	Concentration (equiv./l.)	Temp. (^{o}C)	Duration Time (min)
-2	1.00	2	15.0	20
-1	1.125	3	17.5	30
0	1.250	4	20.0	40
1	1.375	5	22.5	50
2	1.500	6	25.0	60

In most response surface methodology problems, the form of the relationship between the response and the independent variables is unknown. Usually a polynomial of second order models is used. If the fitted surface is an adequate approximation of y then analysis of the fitted surface will be approximately equivalent to analysis of the actual system. The model parameters can be estimated most effectively if proper experimental designs are used to collect the data (Box and George, 1978). Customarily, method of least squares is used to estimate the parameters in the approximating polynomials.

Table (2) Sequence of experiments according to central composite design

Exp. No.	Coded Variable				Real Variable			
	X_1	X_2	X_3	X_4	Current Density (Amp/0.5 dm^2)	Concentration (equiv./l)	Temperature (°C)	Time (min)
1	-1	-1	-1	-1	1.125	3	17.5	30
2	1	-1	-1	-1	1.375	3	17.5	30
3	-1	1	-1	-1	1.125	5	17.5	30
4	1	1	-1	-1	1.375	5	17.5	30
5	-1	-1	1	-1	1.125	3	22.5	30
6	1	-1	1	-1	1.375	3	22.5	30
7	-1	1	1	-1	1.125	5	22.5	30
8	1	1	1	-1	1.375	5	22.5	30
9	-1	-1	-1	1	1.125	3	17.5	50
10	1	-1	-1	1	1.375	3	17.5	50
11	-1	1	-1	1	1.125	5	17.5	50
12	1	1	-1	1	1.375	5	17.5	50
13	-1	-1	1	1	1.125	3	22.5	50
14	1	-1	1	1	1.375	3	22.5	50
15	-1	1	1	1	1.125	5	22.5	50
16	1	1	1	1	1.375	5	22.5	50
17	-2	0	0	0	1.000	4	20	40
18	2	0	0	0	1.5	4	20	40
19	0	-2	0	0	1.25	2	20	40
20	0	2	0	0	1.25	6	20	40
21	0	0	-2	0	1.25	4	15	40
22	0	0	2	0	1.25	4	25	40
23	0	0	0	-2	1.25	4	20	20
24	0	0	0	2	1.25	4	20	60
25	0	0	0	0	1.25	4	20	40
26	0	0	0	0	1.25	4	20	40
27	0	0	0	0	1.25	4	20	40
28	0	0	0	0	1.25	4	20	40
29	0	0	0	0	1.25	4	20	40
30	0	0	0	0	1.25	4	20	40
31	0	0	0	0	1.25	4	20	40

generated by each variable, as well as the interaction effect of the variables reflected on the response.

The present work includes the achievement of experimental work via central composite rotatable designed method to create specimens of different artificial film thickness using sulfuric acid anodizing process. The corrosion resistance, hardness, and roughness of the anodized specimens had been tested to check their confidentiality against untreated specimens of the same alloy. The operating conditions using sulfuric acid anodizing process were commenced with temperature range between 10-25 oC, current density range between 2-3 Amp/dm2, sulfuric acid concentration range between , contact time range between .

The chemical composition for the material according to the analysis of Central Organization for Standardization and Quality Control was listed in Table (4) as follows:

Table (3) Chemical composition of aluminum alloy according to the analysis of central organization and quality control

Cu %	Fe %	Mn %	Zn %	Mg %	Si %	Al %
0.006	0.222	0.037	0.010	2.374	0.100	97.251

The studied material was supplied in a steel form of 30 mm thickness. The sheet is cut into specimens with an overall surface area of 47.2 cm2 (0.472 dm2). Chemical solutions are used as an electrolysis solution, and alkaline, acidic etching solutions as follow: -

1. For anodizing, sulfuric acid solution was used in different normality.
2. For alkaline etching, 5 % NaOH solution was used.
3. For acidic etching, 15 % HNO3 solution was used.

A rectangular box made of polyethylene to resist the action of sulfuric acid was prepared to roll as an anodizing cell. Aluminum weir jigs designed to hold the anodes and the cathodes were fixed in the cell by racks. Direct current power supply (type, mi-dual power supply, TF 2158) was incorporated with the anodizing cell to supply current to the electrodes of maximum of 2 Amp and voltage of 30 volt. To maintain good mixing of the electrolyte solution and to prevent temperature layering in the anodizing cell, a mechanical stirrer (type, Heidolph 50110) was employed. Cooling system was installed which consist of a rectangular cooling bath, Grant instrument (type SU6) for pumping water and an immersed glass coil. Figure (1) shows a schematic diagram for the anodizing apparatus assembly. To heat up the chemicals at the desired temperature through etching and sealing steps a mantel heater was used. Besides, specimen dryer (type MS) was employed for preparing the anodized specimens after oxidation, colouring and sealing steps.

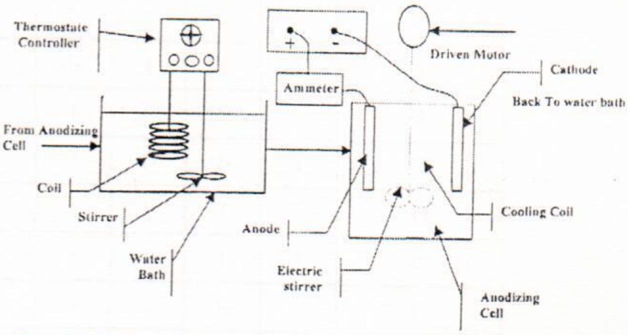


Fig. (1) Schematic diagram for the whole assembly of the anodizing apparatus

Experimental Procedure

Raw material of aluminum-magnesium alloy was received in sheet form. The sheet was cut into small specimens with a dimension of (100x20x3) mm. The edge of the prepared specimens was chamfered by a grinding wheel. Then specimen was etched by dipping in a 5 % NaOH solution (density = 1.06 gm/cm3) for 5 minutes at temperature 40-50 oC. To ensure complete removal of NaOH tap water and distilled water was used. To remove the black layer that was formed and also to activate the surface, the specimen was immersed in 15 % HNO3 solution (density = 1.085 gm/cm3) for 10 seconds at room temperature. Afterward, distilled water and hot air was used for rinsing and drying. At this stage, pretreatment steps before anodizing were completed.

Before ensue the anodizing step the anode and the cathode are well connected to the power supply by aluminum weir jigs. The mechanical stirrer, water bath temperature, concentration of electrolyte and current density were adjusted at pre-designed conditions. After anodizing step (sulfuric acid process), the specimen was rinsed with tap water to remove the residual of electrolyte solution. Sealing the initial porous was performed by immersing the specimen in a flask filled with hot water at 95 oC for 20 minutes. Before sealing the initial porous, colouring of the specimens was commenced by potassium dicromate of 45 gm/l concentration. The colouring step was carried out at 60 oC and lasted for 15 minute. Afterward, drying of the specimen was done by hot air. After each experiment, thickness of the anodized specimen was measured. Thickness-testing meter of type Posi Pilot ®.was used for measuring film thickness.

Studying the corrosion resistance of the specimens was achieved by using weight loss method according to the following equation:

$$\text{mpy} = \text{Mils per Year} = \frac{534W}{DAT} = \text{corrosion rate} \qquad (6)$$

Where W is the weight loss in mg, D is the density of the specimens that determined by Archimedes principle, A is the area of the two faces of specimen in in2, and T is the exposure time in hour.

To prepare a comparison study three uncoloured anodized specimens at optimum conditions, three coloured anodized specimens at optimum conditions and three unanodized specimens are partially immersed in oxidizer solution at static conditions and dynamic conditions. Once again the testing was repeated in fuel solution, and in CASS (i.e. copper-accelerated acetic acid salt-spray test).

The chemical composition for the three types of testing solutions is as follows:

Oxidizer Solution	Composition (%)
N_2O_4	17.5-22.5
HNO_3	Not less than 73.1
HF	0.5-0.7
H_3PO_4	1.0-1.3
H_2O	1.2-2.4
Fuel Solution	**Composition (%)**
Triethylamine	48-52
Xylidin	48-52
H_2O	Max. 0.5

CASS Solution	Composition
Sodium chloride	50±5 g
Cupric chloride	0.26±0.02 g
Water (preferably purified)	1 liter
pH	3.2±0.1 adjusted by the addition of glacial acetic acid

Vickers microhardness test was carried out using LIETZ Optical Microscope (W. Germany). The instrument uses a square-based diamond indenter with angle of 136o between the opposite faces. The magnification was x100 and the applied load was 100 gf. The average of 5 reading of the indentation length was taken while the applied load was kept for 20 sec for each reading. Measuring the surface roughness of the anodized aluminum film was carried out using Talysurf 6 system. The instrument uses tracing stylus where a sharp stylus is drawn over the surface and the vertical displacement is amplified electrically. The arithmetic average value (Ra) for anodized specimen was calculated directly from the instrument. The testing was carried out for coloured, uncoloured and unanodized specimens.

RESULTS AND DISCUSSION

A series of specimens that conducting sulfuric acid anodizing were prepared to study the effect of the most affective variables (i.e. temperature, concentration of electrolyte, time and current density) on anodizing of aluminum-magnesium alloy 5052. These variables had been correlated with the thickness of the specimen by a second order polynomial model. Specimens at the predicted optimum conditions were further manipulated to prepare a final study by measuring the corrosion resistance, hardness and surface roughness for these samples and compare the results with untreated specimens.

The last columns in Table (4) show the experimental and predicted thickness of the anodic film. A second order polynomial correlates the four variables (i.e. the current density, electrolyte concentration, electrolyte temperature and the time of exposure) with the thickness of the anodic film. The best formal of the proposed model, the coded variables, Table (4), were statistically analyized to estimate the coefficients of the proposed model.

Table (4) Values of the experimental and predicted thickness of the oxide film

Exp. No.	Coded Variable				Real Variable				Exp. Thickness	Predicted Thickness
	X_1	X_2	X_3	X_4	Cur. Den. (Amp/0.5dm^2)	Conc. (equiv./l)	Temp. (°C)	Time (min)	Y (Micron)	Y (Micron)
1	-1	-1	-1	-1	1.125	3	17.5	30	3	5.5
2	1	-1	-1	-1	1.375	3	17.5	30	18.1	15.6
3	-1	1	-1	-1	1.125	5	17.5	30	1.5	2.9
4	1	1	-1	-1	1.375	5	17.5	30	14.5	14.0
5	-1	-1	1	-1	1.125	3	22.5	30	5.4	7.6
6	1	-1	1	-1	1.375	3	22.5	30	9.7	6.7
7	-1	1	1	-1	1.125	5	22.5	30	10.3	11.3
8	1	1	1	-1	1.375	5	22.5	30	10.2	11.4
9	-1	-1	-1	1	1.125	3	17.5	50	22.6	21.7
10	1	-1	-1	1	1.375	3	17.5	50	37.1	38.5
11	-1	1	-1	1	1.125	5	17.5	50	3.9	9.3
12	1	1	-1	1	1.375	5	17.5	50	28.9	27.1
13	-1	-1	1	1	1.125	3	22.5	50	21.1	24.0
14	1	-1	1	1	1.375	3	22.5	50	30.8	29.0
15	-1	1	1	1	1.125	5	22.5	50	15.0	17.8
16	1	1	1	1	1.375	5	22.5	50	24.7	24.6
17	-2	0	0	0	1.000	4	20	40	17.8	10.4
18	2	0	0	0	1.5	4	20	40	22.9	27.3
19	0	-2	0	0	1.25	2	20	40	19.7	20.1
20	0	2	0	0	1.25	6	20	40	15.8	12.3
21	0	0	-2	0	1.25	4	15	40	17.3	16.0
22	0	0	2	0	1.25	4	25	40	17.4	15.6
23	0	0	0	-2	1.25	4	20	20	1.2	1.3
24	0	0	0	2	1.25	4	20	60	33.9	30.8
25	0	0	0	0	1.25	4	20	40	16.1	16.1
26	0	0	0	0	1.25	4	20	40	16.1	16.1
27	0	0	0	0	1.25	4	20	40	16.1	16.1
28	0	0	0	0	1.25	4	20	40	16.1	16.1
29	0	0	0	0	1.25	4	20	40	16.1	16.1
30	0	0	0	0	1.25	4	20	40	16.1	16.1
31	0	0	0	0	1.25	4	20	40	16.1	16.1

The coefficients are estimated by implementing nonlinear regression technique. The number of iterations was terminated when the proportion of variance accounted for was equal to 0.9208 and the correlation coefficient (R) was equal to 0.9596. Optimum values were determined using Hooks and Jeeves pattern move technique.

The final form of the proposed model was found as follow: -

$$Y = 16.10006 + 4.224980X_1 - 1.94168X_2 - 0.091682X_3 + 7.366642X_4 + 0.69580X_1^2 + 0.045808X_2^2 - 0.054200X_3^2 - 0.00419X_4^2 + 0.249996X_1X_2 - 2.7500X_1X_3 + 1.662498X_1X_4 + 1.57500X_2X_3 - 2.4625X_2X_4 + 0.037495X_3X_4 \quad (7)$$

Effect of Concern Variables

Equation (7), show thickness dependence of the oxide film on current density (X1), electrolyte concentration (X2), temperature of the electrolyte (X3) and time of exposure (X4) in following sequence: (X4 > X1 > X2 > X3). It shows that the current density (X1) and time of exposure (X4) had a significant dependence on the film growth in comparison with the other two variables, i.e. electrolyte concentration (X2) and temperature (X3). The latter shows the lowest dependence that might regarded in comparison to the rest. Figure (2) shows the dependence of current density on the film thickness at different concentrations, temperatures and times of exposure. The amount of aluminum oxide is directly proportional to the current density. Normally, the thickness of the oxide film increases since current encourages the reaction of the oxygen with aluminum, i.e. to produce aluminum oxide. Statistical analysis of the response function shows large effect of electrolyte concentration (X2) to prohibit film growth in comparison to current density (X1) and time of exposure (X4) (i.e., positive dependencies). Figure (3) shows the effect of concentration on film thickness at different current densities, temperatures and times of exposure. Increasing the concentration causes a drop in film thickness that reaches its lowest value at a concentration of 6N or 25 wt %, which was contributed to greater tendency of film dissolution in higher concentration of sulfuric acid. Temperature effect on oxide film was accounted for in Eq (7), which has negative dependence of smallest value in comparison to the effect of electrolyte concentration. The effect of temperature on film thickness at different current densities, concentrations, time of exposure was monitored in Fig (4). Increasing the temperature of treatment results in a drop in film thickness that reaches its lowest value at temperature 25 oC, which accommodate higher tendency of oxide layer dissolution.

Time of exposure (X4) has a pronounced effect on film thickness in comparison to other variables. This was ascertained from monitoring the increase in film thickness in Fig (5). Obviously, the increase in film thickness is linearly proportional to exposure time within 60 minute of exposure.

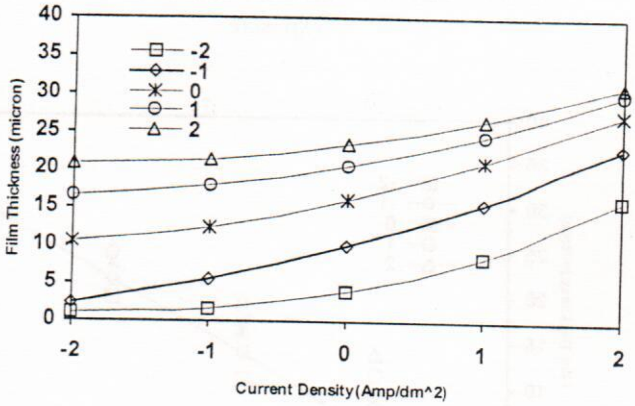
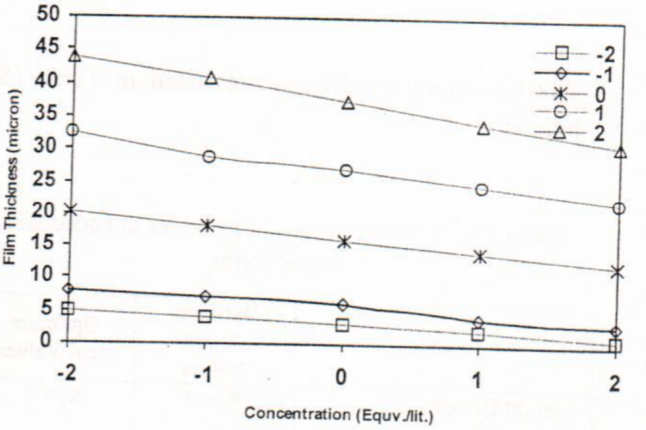


Fig. (2) The effect of current density on film thickness at different concentration, temperature and time of exposure



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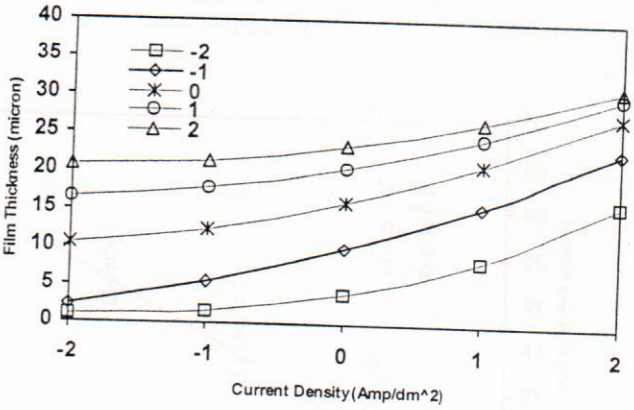

Fig. (2) The effect of current density on film thickness at different concentration, temperature and time of exposure

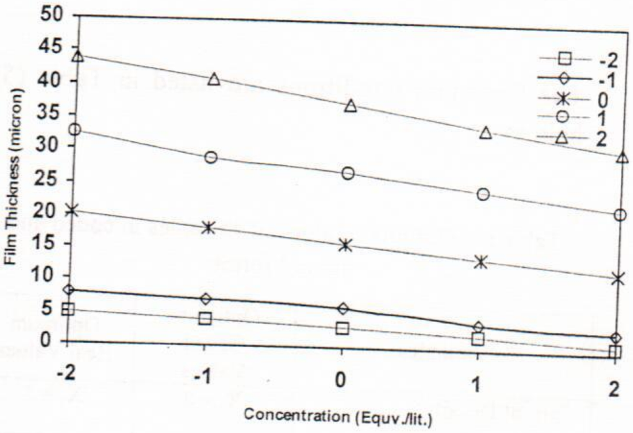

Fig. (3) The effect of concentration on film thickness at different current density, temperature and time of exposure

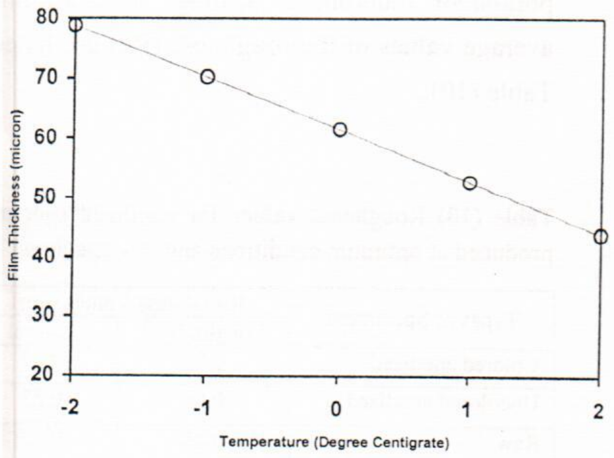

Fig. (8) The effect of temperature on film thickness at optimum conditions (i.e. current density = 1.5 Amp/dm^2, concentration = 2N and time of exposure = 60 min)

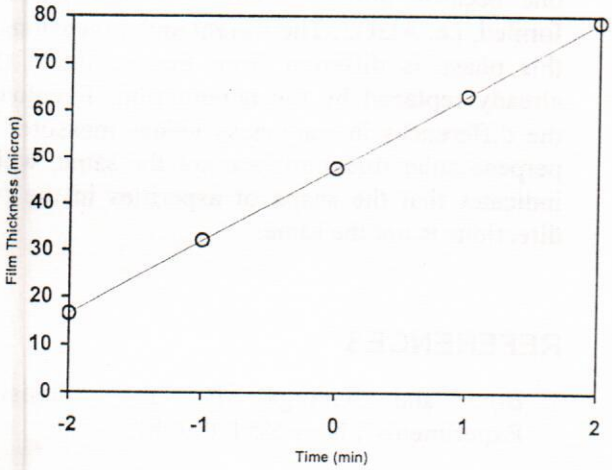

Fig. (9) The effect of time of exposure on film thickness at optimum conditions (i.e. current density = 1.5 Amp/dm^2, concentration = 2N, temperature = 15 °C)

Corrosion Rate Test

Corrosion rates in oxidizer, fuel and CASS solutions are listed in Tables (6), (7) and (8) respectively.

Tables (6) and (7) shows superior corrosion rates in oxidizer solution in comparison to that in fuel solution due to difference in chemical composition of the two solutions. The aluminum tends to react with acidic and alkaline solutions. Since anodized coatings contains Al_2O_3 61.7 % and $Al_2O_3.H_2O$ 7.6 %, the aluminum in this composition tends to react more actively with HF and H_3PO_4 in oxidizer solution than in alkaline amines in fuel solution.

Table (6) Corrosion rates in oxidizer solution

Type of Specimens	Weight Loss (mg)	Corrosion Rate (mil/year)	State of Solution
Colored	258.9	19.58	Static
Colored	284.6	21.53	Dynamic
Uncolored	164.6	12.45	Static
Uncolored	186.4	14.10	Dynamic
Raw	299.0	22.62	Static
Raw Material	317.8	24.04	Dynamic

Table (7) Corrosion rates in fuel solution

Type of Specimens	Weight Loss (mg)	Corrosion Rate (mil/year)	State of Solution
Colored anodized	155.4	11.75	Static
Colored anodized	186.4	14.10	Dynamic
Uncolored anodized	100.4	7.59	Static
Uncolored anodized	133.6	10.10	Dynamic
Raw Material	204.0	15.43	Static
Raw Material	256.0	20.04	Dynamic

Table (8) Corrosion rates in CASS solution

Type of Specimens	Weight Loss (mg)	Corrosion Rate (mil/year)	State of Solution
Colored anodized	45	1.702	Dynamic
Uncolored anodized	25	0.945	Dynamic
Raw Material	89	3.366	Dynamic

Results shows that corrosion rates in dynamic state of solution is superior than that in static state since the reaction rate is accelerated by agitation. Also, the corrosion rates for uncolored specimens are less than from colored specimens since corrosion resistance are largely attributed to the sealing process where sealing in potassium dichromate solution tends to increase the reaction of potassium with acidic or alkaline solutions in oxidizer or in fuel solution. This results in depletion from sealing solution (potassium dichromate) which sealed the anodized surface and inhibit the corrosion rates.

Table (8) listed the corrosion rates of the specimens in a standard testing solution, where aluminum tends to pit in water that containing Cl-, particularly at cervices or at stagnant areas where passivity breaks down through the action of differential aeration cells. Furthermore, traces of Cu+2 (0.1 ppm) in water may react with aluminum that result in depositing metallic copper at local sites. The copper being efficient cathodes, shift the corrosion potential in the noble direction to the critical potential, thereby, both initiating, pitting and by galvanic action stimulating pit growth. Vickers micro-hardness testing results are listed in Table (9).

Table (9) Micro-hardness value for anodized specimens prepared at optimum conditions and raw material specimens

Types of Specimens	Micro-Hardness Values
Colored anodized	125.1
Uncolored anodized	110
Raw	75.2

It was obvious that hardness values of anodized specimens are higher than that of untreated specimens. This refers to the influenced of the existence of aluminum oxide. Generally, hardness decreases with electrolyte temperature, acid concentration. Also, colored specimens shows higher value of hardness in comparison with uncolored specimen. This perhaps was contributed to the variation of chemical composition of traditional coating (Al2O3) by potassium dichromate sealing. The arithmetic average values of the roughness (Ra) are listed in Table (10).

Table (10) Roughness values for anodized specimens produced at optimum conditions and raw specimen

Types of Specimens	Roughness Values (µm)	
	Longitudinal	Lateral
Colored anodized	0.72	0.62
Uncolored anodized	0.58	0.53
Raw	0.43	0.38

The principal reason for the roughness difference between the three specimens is due to size of asperity of each surface. The roughness of anodized surface is greater than that of untreated one because during anodizing a new phase is formed, i.e. Al2O3. The weight and dimensions of this phase is different from that of metal that already replaced by the anodic film. Eventually the differences in roughness values measured in perpendicular directions are not the same, which indicates that the shape of asperities in the two directions is not the same.

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