The Determination of Optimum Conditions for Anodizing Aluminum Alloy (6063).

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Abstract

Aluminum alloy (6063) has been anodized using sulfuric acid as an electrolyte. To study the characteristic of the anodic film, four variables, were considered as the most dominant variables. These variables are: current density in the range of 1- 4 A/dm^2, electrolyte concentration in the range of 6 - 20 vol.%, electrolyte temperature in the range of 10- 30^{o}C and anodizing time between 12- 60 min.

These four variables are manipulated through the experimental work using Box – Wilson experimental design where second order polynomial model was proposed to correlate the studied variables with the thickness of anodic film of aluminum alloy (6063) to estimate the coefficients of the proposed polynomial adopted via statistica software.

The predicated models are found after statistically analyzing the significance as follows:

$$Y = 27.7800 + 8.0737X_1 - 0.8037X_3 + 8.2078X_4 - 0.6994X^2_1 - 0.8882X^2_2 - 1.5582X^2_3 - 1.1231X_1X_2 + 2.6225X_1X_4 - 1.7931X_2X_3 - 1.6956X_2X_4 - 1.0581X_3X_4$$

where Y is the objective function (thickness of anodic film), X_1 is the current density; X_2 is the electrolyte concentration; X_3 is the temperature of electrolyte and X_4 is the anodizing time.

The study shows that the anodizing time and current density had shown positive dependence of great significance on the anodic film thickness while the other two studied variables (i.e. concentration and temperature of electrolyte) had shown small dependence on the film thickness of aluminum alloy (6063).

Optimum conditions for achieving the maximum film thickness are obtained from optimizing the above correlation and are found as follow: 4 A /dm^2 Current density, 6 vol. % Acid concentration, 19.5 ^{o}C Electrolyte temperature and 60 min. time of anodizing.

الخلاصة

يتضمن البحث دراسة أنودة سبيكة الألمنيوم (6063) باستخدام حامض الكبريتيك كمحلول ألكتروليتي . لغرض دراسة خصائص طبقة الانودة لسبيكة الألمنيوم (6063) أُخذت بنظر الأعتبار أربع متغيرات أعتبرت الأكثر تأثيراً على عملية الأنودة.

لتصميم التجارب العملية تم استخدام طريقة (Box- Wilson) لربط المتغيرات أعلاه بمعادلة من الدرجة الثانية تشير الى العلاقة بين المتغيرات الأربعة وسمك طبقة الانودة لسبيكة الألمنيوم(6063) .

من خلال أيجاد المعادلات متعددة الحدود المقترحة والمعتمدة في البرنامج الإحصائي(analysis Statistica) لوحظ بأن النماذج المستنبطة بعد التحليل الإحصائي للعوامل المؤثرة كان كما يلي :

$$Y= 27.7800 + 8.0737X_1 - 0.8037X_3 + 8.2078X_4 - 0.6994X^2_1 - 0.8882X^2_2 -1.5582X^2_3 - 1.1231X_1X_2 + 2.6225X_1X_4 - 1.7931X_2X_3 - 1.6956X_2X_4$$

حيث أن Y تمثل الدالة لسمك طبقة الأنودة, X_1 تمثل كثافة التيار, X_2 تمثل تركيز المحلول الألكتروليتي, X_3 تمثل درجة حرارة المحلول وX_4 تمثل زمن الأنودة.

توضح الدراسة بأن زمن الأنودة وكثافة التيار لها تأثير أيجابي عالي على سمك طبقة الأنودة لسبيكة الألمنيوم(6063) في حين أن المتغيران الآخران (التركيز ودرجة الحرارة) كان لها تأثير محدود على سمك الطبقة. تم أستخراج أفضل ظروف لتنفيذ التجارب من خلال تطبيق المعادلة التجريبية للحصول على أعلى قيمة لسمك الطبقة وهذه الظروف موضحة أدناه: كثافة التيار 4 A/dm^2, تركيز المحلول 6 vol.%, درجة الحرارة 19.5 C° وزمن الانودة 60 min.

KeyWord: Aluminum alloy (6063) , anodizing , sulfuric acid , Box – Wilson experimental design , current density, optimum conditions

1- Introduction

Anodizing is an electrochemical process to produce an oxide film coating on metals or alloys for both protective and decorative purposes. Although many metals can be anodized, aluminum anodizing is the most popular and used in various commercial and military application [(1)].

The coating structures consist of hexagonal pores, which are filled with a seal that hydrolyzes these pores to fill them with inert aluminum oxide [(2)].

Sulfuric acid is by far the most important solution anodizing process due to the characteristics of the process (allows a wide variety of thickness).

Due to its permeable nature, sulfuric anodizing is excellent for clear coating and color dyeing, provides a base for primers, bonding agent and organic coatings. Its provides corrosion resistance and is very durable, typical application manufacturing automotive and computers [(3)].

The 6xxx group is alloyed primarily with both magnesium and silicon and heat treatable to moderate strength, good corrosion resistance, weldability, a unique feature is their great extrudability. Alloy 6063 is perhaps the most widely used because of this feature, its used in architectural applications because of the ease of fabrication by extrusion into shape of great complexity[(4)].

Lin et al. [(5)]showed that the general aluminum buildup rate was significantly increased by using lower current density, higher bath temperature and higher acid concentration, they also showed the chemical dissolution of the anodic coating, in most cases occurring as chemical dissolution of the cell wall, accounts for about 17 to 28% of the total aluminum build-up under anodizing type II conditions (sulfuric acid anodizing by according classification standard MIL-A-8625F (6)), and about 13 to 24% of the total aluminum build-up under anodizing type III conditions (hard coating anodizing by according classification standard MIL-A-8625F (6)), depending on the current density applied.

The aim of this work is to study the formation of anodic oxide film for aluminum alloy (6063) by means of sulfuric acid anodizing process. The effect of the operating conditions (current density, electrolyte concentration, temperature of electrolyte and anodizing time) on film thickness growth for aluminum alloy(6063) under optimum conditions that are obtained by using Box- Wilson techniques .

2- Experimental Design (Box-Wilson design)

Box-Wilson design is a series of tests for characterizing a physical mechanism. These series of experiments have been developed which efficiently serve as a basic deriving the mathematical model of process (7). For four variables, the quadratic polynomial equation can be represented as follows:

$$Y= B_0+ B_1X_1+ B_2X_2+ B_3X_3+ B_4X_4+ B_{11}X_1^2+ B_{22}X_2^2+ B_{33}X_3^2+ B_{44}X_4^2 + B_{12}X_1X_2+B_{13}X_1X_3+B_{14}X_1X_4+B_{23}X_2X_3+ B_{24}X_2X_4+B_{34}X_3X_4 \quad \ldots\ldots\ldots (1)$$

A preliminary step is to set up the relationships between the coded level and the corresponding real variables, which are required in the determination of experimental range by the following equation.

$$Xcoded = \frac{Xactual - Xcenter}{\dfrac{Xcenter - X\min}{\sqrt{k}}} \quad ..(2)$$

The coded variables take the value between -2 and 2 in accordance with the central composite rotatable suggested by Cochrun (8).

The range of real variables for the system could be represented in Table (1).

The expression of coded level for the system is estimated from equation (2).

$$X_1 = \frac{A-2.5}{0.75} \quad \ldots\ldots\ldots(3)$$

$$X_2 = \frac{C-13}{3.5} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots(4)$$

$$X_3 = \frac{T-20}{5} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

$$X_4 = \frac{t-36}{12} \quad \ldots\ldots\ldots\ldots\ldots\ldots(6)$$

where :-

A = current density (A / dm^2)

C = concentration of electrolyte (vol %)

T = temperature of electrolyte (^{o}C).

t = time of anodizing (min.).

Table (2) shows the relationship between the coded level and corresponding real variables.

The number of experiments that must be done shown in Table (3).

3- Experimental Work

3-1 Materials

3-1-1 Anode

The materials used in this study is aluminum alloys (6063) .This material is cut of specimens by dimensions of (2*1*0.07) cm .

Analysis of this specimens was carried out using (spark technique). Table (4) shows the nominal and the analytical chemical compositions of material used in this work. This analysis indicates that the main elements of material is within the standard limits.

3-1-2 Cathode

Aluminum alloy (6063) with dimensions (8*4*0.07) cm was used.

3-2 Experimental Procedure

3-2-1 Pre-Treatment

Prior to anodizing, the specimen was treated with the following processes:

3-2-2 Mechanical Pre-Treatment

Raw materials of pure aluminum and aluminum alloy were received in the form of sheet. The sheet was cut into small specimens with dimensions previously mentioned where sharp edges and then chamfered via grinding wheel.

3-2-3 Chemical Pre-Treatment

(I) Oil, grease and general dirt where properly removed with acetone at 25°C. Grease tends to float on surface, which was removed later by filtration. After this stage, the specimen was rinsed in running water then in distilled water to remove the excess of acetone on the specimen.

(II) Alkaline Etching**:** The specimen was etched by dipping in 5% volume sodium hydroxide solution for 5 minutes at temperature of 45°C , the the specimen was rinsed by tap water and then by distilled water to ensure the removal of the excess solution of sodium hydroxide.

(III) Desmutting: The specimen was treated in 5% nitric acid solution for about 5 minutes at 25°C to remove the black layer that formed on the surface and to activate the surface for the anodizing stage. After ward, the specimen was rinsed with running water followed by distilled water and dried by means of air.

(IV) Electropolishing: The specimen was polished by using the electro- polishing technique by immersion in the solution consists of :

Phosphoric acid	75%
Sulfuric acid	25%
Nitric acid	0.1%

The temperature of electrolyte is 90°C and the time immersion is 10 min and the current density is 15 A/dm^2 . The specimen was rinsed with running water after electropolishing followed by distilled water and dried, then kept in the desiccator over a silica gel before being weighed (W1).

3-3 Anodizing

3-3-1 Operating Procedure

The following steps were followed in this stage:

1- After pretreatment, weighing specimens before anodizing (W_1).

2- The solution of desired acid concentration (sulfuric acid) was placed in anodizing cell.

3- The thermostat was set to the desired operating temperature.

4- When all the requirements of experiment were set up, the power supply was switched on a fixed constant current at the desired value. The electrodes were immersed in the solution while the power supply was switched on, in which voltage increased gradually.

5- At the end of the desired time, the power supply is switched off and the anode was removed from anodizing cell immediately.

6- After the specimen was removed from anodizing cell, it rinsed with running water, followed by distilled water to remove the excess solution on the specimen.

7- The specimen was dried in a drying furnace at 50°C for 30 minutes and stayed in desiccator about 30 minutes.

8- The specimen was weighed after anodizing (W_2).

Figure (1) shows a schematic diagram for the whole assembly of the anodizing apparatus .

3-3-2 Stripping Anodic Coating

Anodic film was stripped using the following solution:

Phosphoric acid	35 ml
Chromic acid	20 g
Distilled water	liter

The temperature of electrolyte is 100°C, and the time for immersion is 10 minutes. After stripping, the specimens were rinsed with running tape water followed by distilled water to remove the excess solutions, then dried in a drying furnace at 50°C for 30 minutes and stayed in desiccator about 30 minutes and weighed (W_3).

3-3-3 Coating Thickness Test

The coating thickness can be calculated from the equation (7) [10,11] :

$$Y = \frac{(W_2 - W_3)\times 10^4}{A\times\rho} \quad \text{..............(7)}$$

where:

$Y =$ coating thickness in micron.

$W2 =$ weight of a sample with anodic coating in (g).

$W3 =$ weight of a sample after stripping in (g).

$A =$ surface area (dm^2).

$\rho =$ density in g/cm^3 is about 2.4 for unsealed coating.

4- Results and Discussion

Table (5) will be first fitted through nonlinear regression analysis to estimate the coefficients of the proposed model. Table (5) also shows the thickness of the oxide films of the specimens that reached through the experimental work and the predicated thickness, which are designed according to the central composite rotatable design method.

4-1 Estimation the Coefficient of the Proposed Model

Using the coded data of the central composite design, Table (6) the coefficients of the 2nd order polynomial were estimated by implementing nonlinear regression estimation technique via the Statistica software.

4-2 Analysis for Significant Values

From Table (5)

$e_i = (Y - y)$

An estimation of the experimental error variance (S_r^2) is obtained by dividing the residual sum of squares (e_i) by number of degree of freedom (γ); where:

$$\gamma = N_i - N_{coff} \quad \text{....... (8)}$$

where:

Ni: No. of experiments

N_{coff}: No. of coefficients in the model

$$\gamma = 28 - 15 = 13 \quad \ldots\ldots (9)$$

$$S_r^2 = \Sigma e_i^2 / \gamma \quad \ldots\ldots (10)$$

The estimated variance of coefficients (S_b^2) are then calculated by the following formula:

$$S_b^2 = S_r^2 / \Sigma X^2 \ldots\ldots (11)$$

The significant coefficient can be estimated by comparing the value of (B^2/ S_b^2) to the critical value $F_{0.95\ (1,\ 13)} = 4.67$ of the F –distribution at 95% level of confidence (12). The results of these calculations are shown in Tables (7) for aluminum alloy (6063).

the final form of the proposed model was as follows:

$$Y = 27.7800 + 8.0737X_1 - 0.8037X_3 + 8.2078X_4 - 0.6994X^2_1 - 0.8882X^2_2 - 1.5582X^2_3 - 1.1231X_1X_2 + 2.6225X_1X_4 - 1.7931X_2X_3 - 1.6956X_2X_4 - 1.0581X_3X_4 \quad \ldots\ldots (12)$$

4-3 Estimating the Optimum Conditions

According to Equations (12), using Hook and Jeeves pattern conditional method in terms of maximum thickness of the anodic film, the optimum conditions were obtained. The optimum conditions of the studied variables in coded and real form are listed in Table (8) below for aluminum alloy (6063).

4-3 Effect of Concern Variables

Figure (2) shows the effect of current density on film thickness for aluminum alloy (6063). the thickness of aluminum oxide is directly proportional to the current density. Normally, the thickness of the oxide film increases to a maximum value at which current encourages the reaction of the oxygen with aluminum, i.e. to produce aluminum oxide.

Figures (3) shows the effect of concentration on film thickness for aluminum alloy (6063). It noticed that the lowest film thickness value is at a concentration of 20 Vol.%, which contributes to the great tendency of film

Figures (3) shows the effect of concentration on film thickness for aluminum alloy (6063). It noticed that the lowest film thickness value is at a concentration of 20 Vol.%, which contributes to the great tendency of film dissolution in higher concentration of sulfuric acid causes decrease in film thickness.

The effect of temperature on film thickness in optimum conditions is shown in Figure (4) for aluminum alloy (6063). Increasing the electrolyte temperature results in a high film thickness that reaches at a counter balance of 19.5 C° where the dissolution rate equal of the formation rate, after this point the dissolution is higher and thickness film is decreased.

Figure (5) shows the effect of time on film thickness for aluminum alloy (6063).

Time of anodizing has a pronounced effect on film thickness in comparison with other variables. Figures (5) shows increase in film thickness for aluminum alloy (6063). It is obviously observed that the increase in film thickness is linearly proportional to anodizing time.

Basheer (13) indicates in his study that the time of anodizing and current density have positive dependence of great significance on the anodic film thickness of pure aluminum and aluminum alloy (3003) . Khalid (14) proved that the film thickness increases in aluminum alloy (6061) with an increase in current density.

Also, Sarmad (15) points out that an increase in concentration makes a drop in film thickness.

5- Conclusions

The second order polynomial regression analysis of the objective function (thickness) in terms of four variables (i.e., current density, concentration of electrolyte, temperature and time of anodizing) gives equation (12), which adequately describes the behavior of the process throughout the studied range.

The four variables affect the film growth in following order:

Anodizing time > Current density > Concentration > Temperature.

The optimum conditions as predicted from equation (4) is 4 A / dm^2 current density, 6 vol. % electrolyte concentration , 19.5^{o}C electrolyte temperature and 60 minute of anodizing time for Al-alloy 6063.

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Table (1) The experimental range of variables.

Current density (A / dm^2)	Concentration (vol) %	Temperature (^{o}C)	Time (min.)
1-4	**6-20**	**10-30**	**12-60**

Table (2) The relationship between coded code level and corresponding real variable.

Variables	**Levels**				
X_1 , X_2 , X_3 ,X_4	**-2**	**-1**	**0**	**1**	**2**
X_1= Current Density (A/dm^2)	1	1.75	2.50	3.25	4
X_2= Concentration (vol %)	6	9.5	13	16.5	20
X_3= Temperature (^{o}C)	10	15	20	25	30
X_4= Time (min.)	12	24	36	48	60

Table (3) Sequence of experiments according to control composite design.

Exp. No.	Coded variables				Real variables			
	X1	X	X3	X4	Current Density (A/dm^2)	Conc. (vol %)	Temp. (°C)	Time (min.)
1	-1	-1	-1	-1	1.75	9.5	15	24
2	1	-1	-1	-1	3.25	9.5	15	24
3	-1	1	-1	-1	1.75	16.5	15	24
4	1	1	-1	-1	3.25	16.5	15	24
5	-1	-1	1	-1	1.75	9.5	25	24
6	1	-1	1	-1	3.25	9.5	25	24
7	-1	1	1	-1	1.75	16.5	25	24
8	1	1	1	-1	3.25	16.5	25	24
9	-1	-1	-1	1	1.75	9.5	15	48
10	1	-1	-1	1	3.25	9.5	15	48
11	-1	1	-1	1	1.75	16.5	15	48
12	1	1	-1	1	3.25	16.5	15	48
13	-1	-1	1	1	1.75	9.5	25	48
14	1	1-	1	1	3.25	9.5	25	48
15	-1	1	1	1	1.75	16.5	25	48
16	1	1	1	1	3.25	16.5	25	48
17	-2	0	0	0	1	13	20	36
18	2	0	0	0	4	13	20	36
19	0	-2	0	0	2.5	6	20	36
20	0	2	0	0	2.5	20	20	36
21	0	0	-2	0	2.5	13	10	36
22	0	0	2	0	2.5	13	30	36
23	0	0	0	-2	2.5	13	20	12
24	0	0	0	2	2.5	13	20	60
25	0	0	0	0	2.5	13	20	36
26	0	0	0	0	2.5	13	20	36
27	0	0	0	0	2.5	13	20	36
28	0	0	0	0	2.5	13	20	36

Table (4) A Nominal (9) and Analytical chemical composition of Al alloy (6063).

Metal	Al	Si	Fe	Cu	Mn	Mg	Cr	Zn	Other
Nominal	Rem.	0.2-0.6	0.35	0.1	0.1	0.45-0.9	0.1	0.1	0.15
Analytical	Rem.	0.5	0.3	0.1	0.1	0.79	0.2	0.1	0.12

Table (5) Values of the experimental measured thickness for aluminum alloy (6063)

Exp. No.	Coded variable				Real variable				Exp. thickness	Predicted thickness	Corresponding residual
	X1	X2	X3	X4	Current Density (A /dm^2)	Conc. (vol%)	Temp (^{o}C)	Time (min.)	Y(μm)	y(μm)	e_i=Y-y
1	-1	-1	-1	-1	1.75	9.5	15	24	6.22	4.327	1.892
2	1	-1	-1	-1	3.25	9.5	15	24	23.09	20.739	2.350
3	-1	1	-1	-1	1.75	16.5	15	24	12.16	13.154	-0.994
4	1	1	-1	-1	3.25	16.5	15	24	25.07	25.072	-0.029
5	-1	-1	1	-1	1.75	9.5	25	24	9.23	9.439	-0.209
6	1	-1	1	-1	3.25	9.5	25	24	23.00	23.817	-0.817
7	-1	1	1	-1	1.75	16.5	25	24	11.59	11.092	0.497
8	1	1	1	-1	3.25	16.5	25	24	22.20	20.979	1.220
9	-1	-1	-1	1	1.75	9.5	15	48	23.47	23.252	0.217
10	1	-1	-1	1	3.25	9.5	15	48	45.06	45.661	-0.601
11	-1	1	-1	1	1.75	16.5	15	48	26.01	25.296	0.713
12	1	1	-1	1	3.25	16.5	15	48	44.86	43.212	1.647
13	-1	-1	1	1	1.75	9.5	25	48	24.03	24.131	-0.101
14	1	-1	1	1	3.25	9.5	25	48	46.94	44.507	2.432
15	-1	1	1	1	1.75	16.5	25	48	18.09	19.002	-0.912
16	1	1	1	1	3.25	16.5	25	48	32.89	34.886	-1.996
17	-2	0	0	0	1	13	20	36	8.95	8.834	0.115
18	2	0	0	0	4	13	20	36	39.68	41.129	-1.449
19	0	-2	0	0	2.5	6	20	36	22.71	24.624	-1.914
20	0	2	0	0	2.5	20	20	36	24.41	23.829	0.580
21	0	0	-2	0	2.5	13	10	36	21.21	23.154	-1.944
22	0	0	2	0	2.5	13	30	36	20.55	19.939	0.610
23	0	0	0	-2	2.5	13	20	12	8.67	9.971	-1.301
24	0	0	0	2	2.5	13	20	60	42.77	42.802	-0.032
25	0	0	0	0	2.5	13	20	36	27.80	27.780	0.199
26	0	0	0	0	2.5	13	20	36	27.90	27.780	0.119
27	0	0	0	0	2.5	13	20	36	27.52	27.780	-0.260
28	0	0	0	0	2.5	13	20	36	27.90	27.780	0.119

Table (6) the coefficient values of the predicted correlation.

Coeff..	Bo	B1	B2	B3	B4	B11	B22	B33	B44	B12	B13	B14	B23	B24	B34
Value	27.78	8.07	-0.19	-0.80	8.20	-0.69	-0.88	-1.55	-0.34	-1.12	-0.50	2.62	-1.79	-1.69	-1.05
Correlation Coefficient(R)					0.9943			Variance explained (%)						98.879	
Final value of loss function					38.9847										

Table (7) Analysis of variance of coefficients effect of aluminum alloy (6063).

Constant Estimated	$\sum X^2$	Estimated Coefficient(B)	Variance $Sb^2=Sr^2/\sum X^2$	F-value $=B^2 / S_b^2$	$F_{0.95\ (1,13)} = 4.67$
B1	24	8.0737	0.1250	521.4	S
B2	24	-0.1987	0.1250	0.316	NS
B3	24	-0.8037	0.1250	5.168	S
B4	24	8.2078	0.1250	538.9	S
B11	48	-0.6994	0.0625	7.828	S
B22	48	-0.8882	0.0625	12.62	S
B33	48	-1.5582	0.0625	38.85	S
B44	48	-0.3482	0.0625	1.940	NS
B12	16	-1.1231	0.1875	6.727	S
B13	16	-0.5081	0.1875	1.376	NS
B14	16	2.6225	0.1875	36.67	S
B23	16	-1.7931	0.1875	17.14	S
B24	16	-1.6956	0.1875	15.33	S
B34	16	-1.0581	0.1875	5.971	S

Table (8) Optimum conditions in coded and real values.

Variables	Opti. conditions (alloy - 6063)	
	Coded	Real
Current Density (A/dm^2)	2	4
Electrolyte Concentration (vol%)	-2	6
Electrolyte Temperature (^{o}C)	-0.110	19.5
Anodizing Time (min.)	2	60
Function Maximum (Film thick.μm)	74.78	

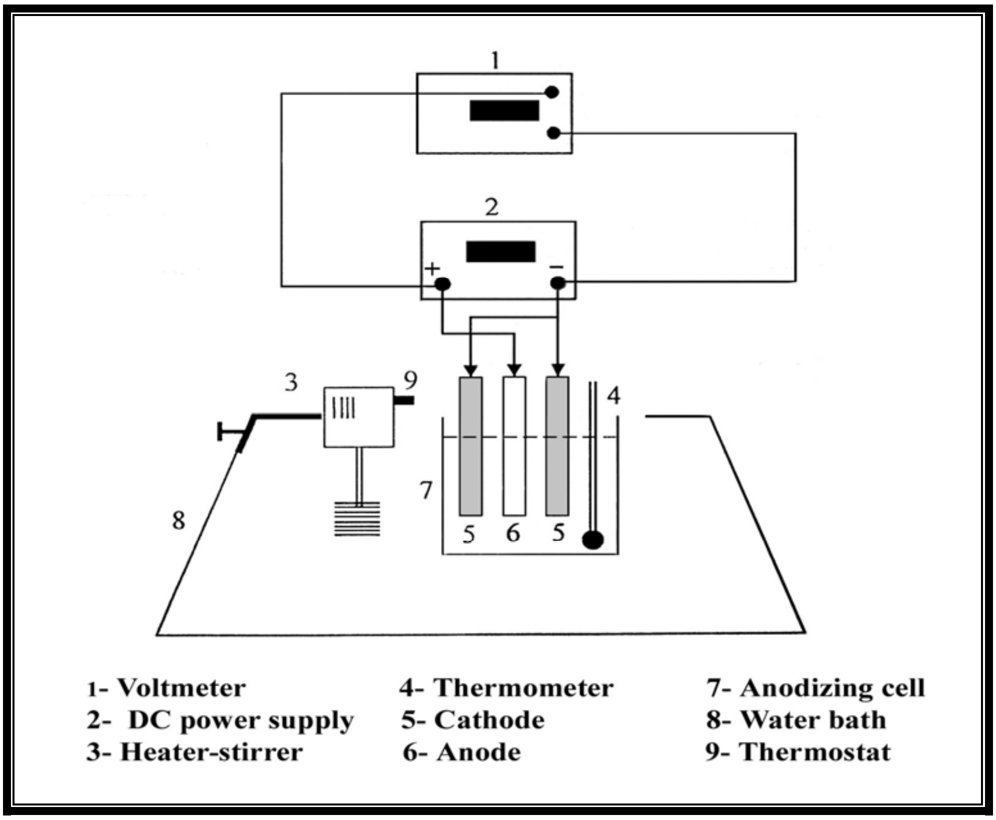
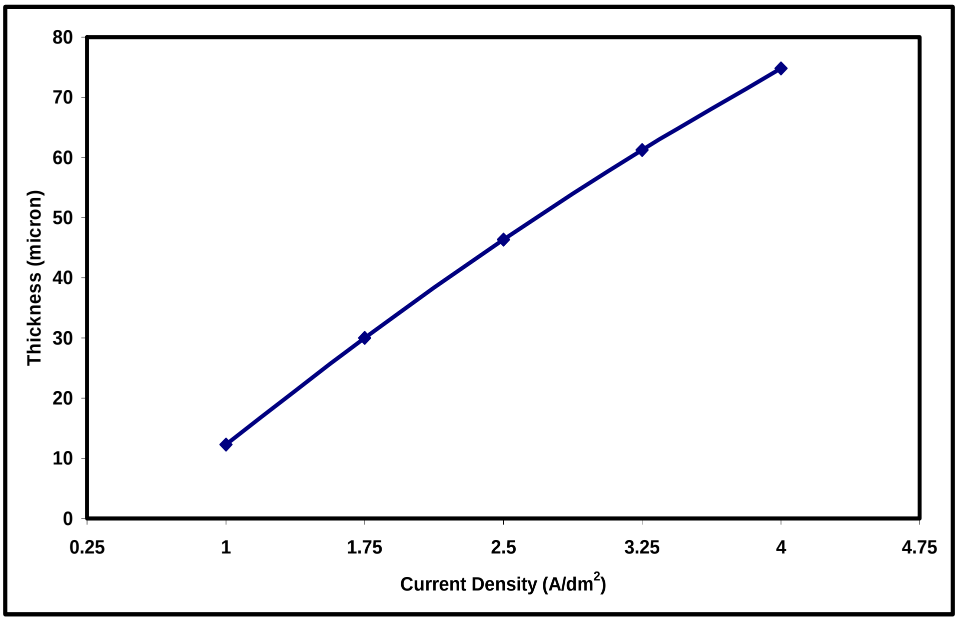
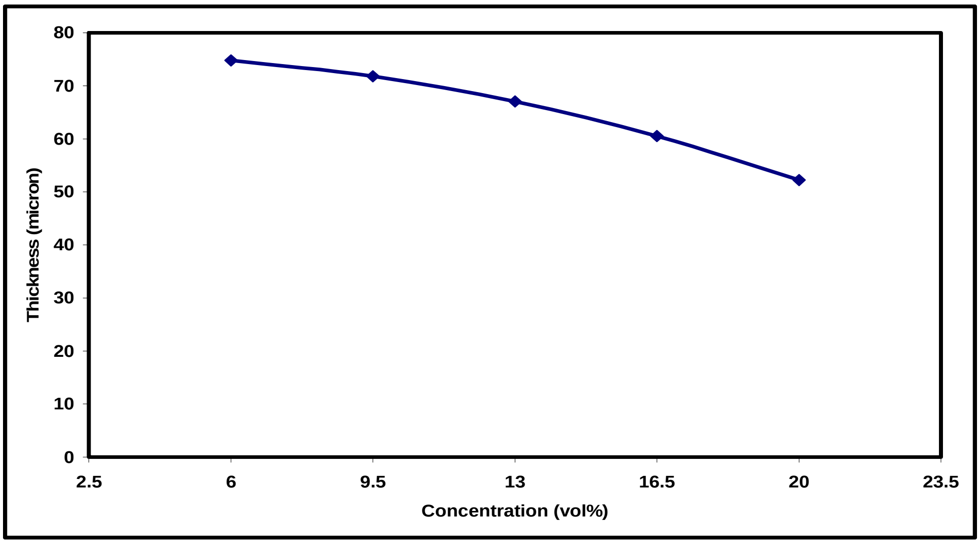


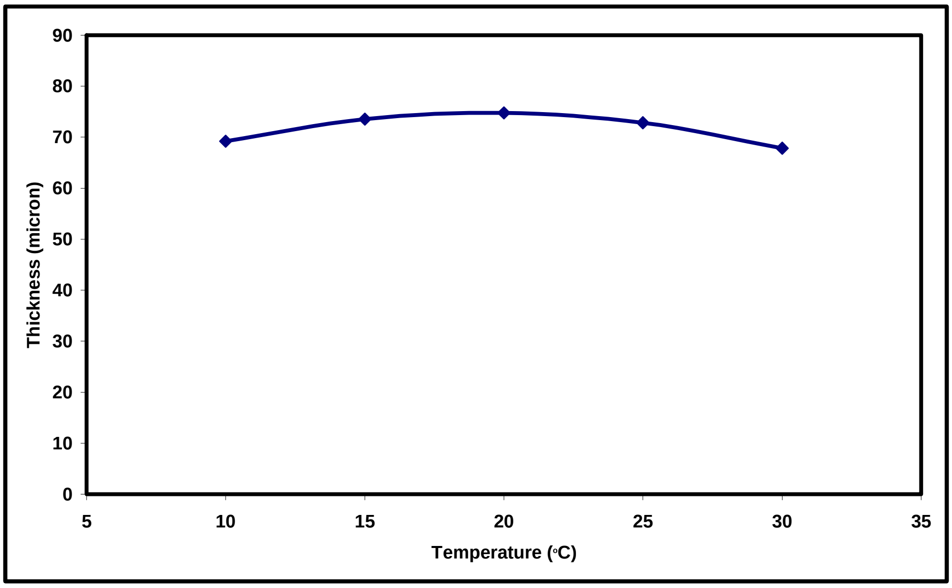
Figure (1) schematic diagram for the whole assembly of the anodizing apparatus.



Figure(2)Effect of current density on film thickness at optimum conditions of Al alloy (6063).



Figure(3)Effect of concentration on film thickness at optimum conditions of Al alloy (6063).



Figure(4)Effect of temperature on film thickness at optimum conditions of Al alloy (6063).

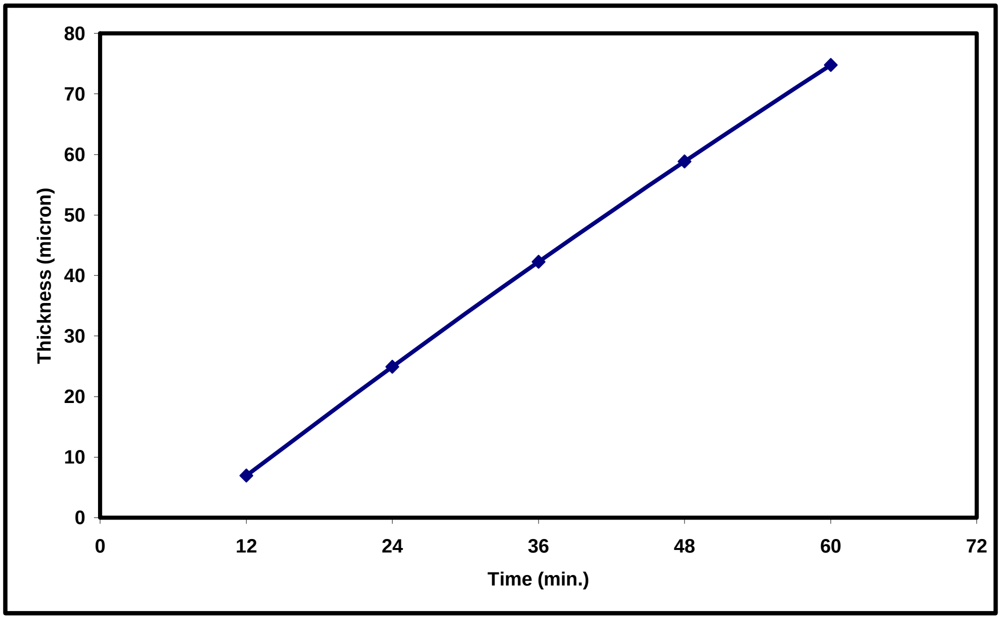


Figure (5) Effect of time on film thickness at optimum conditions of Al alloy (6063).