

Optimization of Mild Steel Anodizing Using Box-Wilson Experimental Design

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ABSTRACT

In this study, a mild steel alloy has been used to determine the influences of voltage, temperature, concentration and time on the film growth, thickness, characteristics and properties of anodic oxide film. A Box–Wilson experimental design method was used to determine effects of parameters and optimum conditions for anodized mild steel alloy. Anodizing of the mild steel alloys in potassium hydroxide solution generates the barrier and porous layers of anodic films according to the conditions used. Mild steel alloy is anodized under different conditions in the KOH solution. Constant voltage technique is used to form oxide films on mild steel alloys in (KOH) electrolyte as a function of the anodization voltage of (1-6V), concentration of (40-70Vol. %), temperature of (20-100°C) and anodization time of (5-75 min). The second order response surface model is given in equations. The model is used in the formulation of objective and constraint functions for the optimization of a optimum conditions of mild steel:

$$T=2.949+0.781X_1+0.131X_2+1.240X_3+0.334X_4+0.047X_{11}+0.003X_{22}+0.377X_{33}-0.153X_{44}+0.041X_{12}+0.115X_{13}+0.026X_{14}+0.046X_{23}+0.025X_{24}+0.141X_{34}$$

Optimum conditions for anodizing mild steel in the alkaline solution were determined by Hook and Jeeves method. The optimum conditions were found as follows: voltage (1V), KOH concentration (40Vol.%), temperature (20°C), and time (5 min). The thickness for mild steel alloy was [0.65µm]. The electrical conductivity characteristic of the oxide was measured; it was found that it has electrical conductivity but with different values depending on anodization conditions. Different colors of anodic oxide film are formed on carbon steel alloys and change with different conditions. The pores diameter for mild steel was [91.78 nm]. AFM topography shows the roughness values of mild steel is [0.496 nm]. The microhardness of anodic oxide film was [1477.5 Hk].

ايجاد الظروف المثالية لانودة الصلب الكربوني المطاوع باستخدام تصميم تجريبي بطريقة (Box-Wilson)

الخلاصة

استخدمت في هذه الدراسة سبيكة فولاذ طري لتحديد تأثير كل من الجهد المسلط , درجة الحرارة , التركيز والزمن على نمو الطبقة , السمك , خصائص وصفات طبقة اوكسيد الانودة. تم انودة هذا الفولاذ الطري في محلول هيدروكسيد البوتاسيوم لتكوين طبقة الانودة المتألفة من طبقة حاجزة وطبقة مسامية اعتمادا على الظروف المستخدمة. يتم انودة الفولاذ الطري بظروف مختلفة في محلول هيدروكسيد البوتاسيوم (KOH). وتتم عملية الانودة باستخدام تقنية الجهد المسلط الثابتة لتشكيل طبقة اوكسيد على سطح سبائك الفولاذ الطري في المحلول الالكتروليتي أعلاه , جهد الانودة الثابت كان من (1-6V) , التركيز من (40-70Vol.%), درجة الحرارة من (20-100°C) وزمن الانودة من (5-75min). ولغرض تصميم التجارب العملية تم استخدام طريقة (Box-Wilson) لربط المتغيرات اعلاه بمعادلة من الدرجة الثانية تشير الى العلاقة بين المتغيرات الاربعة وسمك طبقة الانودة المتكونة على سطح الصلب الكربوني الخامد والطري من خلال ايجاد ثوابت للمعادلات المتعددة الحدود. المعادلة التي تم الحصول عليها كانت كما يلي:

$$T=2.949+0.781X1+0.131X2+1.240X3+0.334X4+0.047X11+0.003X22+0.377X33-0.153X44+0.041X12+0.115X13+0.026X14+0.046X23+0.025X24+0.141X34$$

تم تحديد الظروف المثالية لانودة الصلب الطري باستخدام برنامج (Hook - Jeeves) للحصول على امثل سمك لطبقة الانودة وكانت الظروف كما يلي: (1V, 40Vol.%, 20°C, 5min). سمك الطبقة كانت (0.65µm) فحصت الموصلية الكهربائية للطبقة الاوكسيدية ووجد انها موصلة للكهربائية وتعتمد على ظروف الانودة. وجد بان الموصلية الكهربائية للطبقة تتغير بقيم مختلفة تعتمد على ظروف الانودة. تظهر الوان مختلفة لطبقات الانودة الاوكسيدية على سبائك الصلب الكربوني والتي تتغير بتغير ظروف الانودة المختلفة. قطر المسام للصلب الطري كان (91.78nm). من خلال طوبوغرافية سطح العينات بجهاز (AFM) لوحظ ان قيم خشونة للصلب الطري كان (0.496nm) مقارنة بالعينه قبل الانودة. من خلال فحص الصلادة الدقيقة (microhardness) للسبيكة المؤنودة بالظروف المثالية تم ملاحظة زيادة الصلادة للصلب الطري وكانت القيمة (1477.5HK).

INTRODUCTION

The mild steel is low cost material, easy availability and excellent physical properties, it is also cheaper than wrought iron and stronger and more workable than cast iron. These properties make it is one of the best preferred materials for industry, but its use is restricted in acidic environments because of the susceptibility towards corrosion. Due to bare steels rust when exposed to neutral pH water, saltwater, or high humidity air and required to improve corrosion resistance and wear resistance. There are many methods to modify metal surfaces, these methods considered advancements in the field of surface engineering for protecting metals and alloys from corrosion such as Conversion coatings, passivation, electro polishing, and anodizing. These methods involve the formation of barrier layer which can be a sacrificial coating, such as zinc or aluminum, or an organic coating, such as paints, waxes or oils, or a barrier oxide [1,2,3]. Anodization process consists artificial growth of oxide layer from the substrate, leading to better adhesion compared to coating process where other material is deposited on the substrate [4].

The oxide film formed in passivation usually has a denser structure and higher chemical stability than bulk iron, therefore tends to mitigate further iron oxidation by preventing the direct contact between bulk iron and oxygen, hydroxyls and/or water molecules. Therefore, the multi-layer iron oxide film on steel surface is commonly referred to as the “protective layer” to reduce corrosion rate. There are many attempts using electric current and an electrolyte to electrochemically form a protective oxide on steel [5].

The anodic film thus formed is composed of a compact inner layer and a porous outer layer and due to this porous structure, anodic film is susceptible to aggressive environments. Therefore, this porous anodic film is sealed by different methods like boiling water, nickel acetate and dichromate solutions to further improve the corrosion resistance [4]. Magnetite (Fe_3O_4) is one of the most important magnetic materials and is widely used in industry [6]. This layer can also act as an effective physical barrier that protect against corrosion.

Carbon steel material can grow a passive oxide film in caustic solutions. The protective oxides on steels during atmospheric exposure for several years form an outer red rust layer, and an inner black protective layer. The inner black layer has been identified by Raman spectroscopy as magnetite (Fe_3O_4) and hematite (Fe_2O_3) [2]. The protective nature of the layer, which formed on the surface of metal under particular form of oxidation is supposed to be connected with the crystal structure of the passive film. Thus, anodic passivity of metals is resulted from the formation of oxide or hydroxide films in the reactions of metals with water. The anodic passive film as suggested by Chao et.al. contains a high concentration of point defect such as metal vacancies, electrons and holes [7]. Depending on the extent of iron oxidation, the iron oxide layer formed on steel surface could have different levels of thickness, from a few angstroms to some tens of nanometers [5].

This work is to investigate the formation of anodic oxide film for mild steel by means of potassium hydroxide (as alkaline electrolyte) anodizing process with constant voltage; in terms of applied voltage, electrolyte concentration, temperature of electrolyte, and time of anodizing in order to study the effect of these four variables on film thickness growth for mild steel.

Experimental Design (Box-Wilson design)

Box and Wilson (1951) are introduced a new method for search the optimal conditions of chemical reactions on the base of modern mathematical statistics by experimental design and data analysis [8]. Box and Wilson (1951) lay the basic foundations for response surface methodology (RSM), which is an integration of experimental design, regression, and optimization theory. RSM is widely used to explore and to optimize response surfaces in industrial experiments [9].

Then a mathematical model in the form of a second-order polynomial is formed. When describing a response surface by a second-order equation, varying a factor on only two levels does not offer the necessary information. Hence, an experiment is designed so that factors are varied on three or more levels. One of such designs is the second-order rotatable design (Box-Wilson design) [10].

$$Y = B_0 + \sum_{i=1}^k B_i X_i + \sum_{i=1}^k B_{ii} X_i^2 + \sum_{i < j} \sum_{j=1}^n B_{ij} X_i X_j + \varepsilon \quad \dots (1)$$

Where:

ε is a random error component and Y is the objective function and k_i is the number of variables in the system.

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 as shown in the table (1).

The coded level in Table (2) used to represent difference values of minimum, medium and maximum to corresponding real variables. Table (3) shows the number of experiments that must be done and gives the relationship between coded variables and real variables.

$$X_{coded} = \frac{X_{actual} - X_{center}}{X_{center} - X_{min}} \frac{\sqrt{k}}{\dots} \quad \dots (2)$$

Table (1) The expression equations used to estimate coded level for the system.

Variables	Equations
Voltage (V)	$X_1 = \frac{V - 3.5}{1.25}$
Concentration (Vol. %)	$X_2 = \frac{C - 55}{7.5}$
Temperature (°C)	$X_3 = \frac{T - 60}{20}$
Time (min.)	$X_4 = \frac{t - 40}{17.5}$

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

Variables	Levels				
	-2	-1	0	1	2
X_1, X_2, X_3, X_4					
$X_1 =$ Voltage (V)	1	2.25	3.5	4.75	6
$X_2 =$ Concentration (Vole.%)	40	47.5	55	62.5	70
$X_3 =$ Temperature (°C)	20	40	60	80	100
$X_4 =$ Time (min.)	5	22.5	40	57.5	75

Experimental Work

Materials

Anode and Cathode

The materials used in anodizing process are mild steel alloys as anode specimens. These materials are cut into sheet specimens of dimensions (30*20*5) mm. The cathode was semi-circle of stainless steel sheet type 304 with dimension of (70*117*1) mm. The potentials were applied using power supply types(RXN3020D,ziAOXINELECTRONICSCO.,LTD, china at maximum of 20A and voltage of 30V) connected with the anodizing cell to supply constant voltage to the anode and cathode electrodes.The space between two electrodes was (50mm) [2]. The ratio of anode to cathode area was (1:5) [11].

Table (4) shows the chemical analysis with the standard chemical compositions value for mild steel alloy used in this work.

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

Exp. No.	Coded variables				Real variables			
	X ₁	X ₂	X ₃	X ₄	Volt. (V)	Conc. (Vol. %)	Temp. (°C)	Time (Min.)
1	-1	-1	-1	-1	2.25	47.5	40.0	22.5
2	1	-1	-1	-1	4.75	47.5	40.0	22.5
3	-1	1	-1	-1	2.25	62.5	40.0	22.5
4	1	1	-1	-1	4.75	62.5	40.0	22.5
5	-1	-1	1	-1	2.25	47.5	80.0	22.5
6	1	-1	1	-1	4.75	47.5	80.0	22.5
7	-1	1	1	-1	2.25	62.5	80.0	22.5
8	1	1	1	-1	4.75	62.5	80.0	22.5
9	-1	-1	-1	1	2.25	47.5	40.0	57.5
10	1	-1	-1	1	4.75	47.5	40.0	57.5
11	-1	1	-1	1	2.25	62.5	40.0	57.5
12	1	1	-1	1	4.75	62.5	40.0	57.5
13	-1	-1	1	1	2.25	47.5	80.0	57.5
14	1	-1	1	1	4.75	47.5	80.0	57.5
15	-1	1	1	1	2.25	62.5	80.0	57.5
16	1	1	1	1	4.75	62.5	80.0	57.5
17	-2	0	0	0	1.00	55.0	60.0	40.0
18	2	0	0	0	6.00	55.0	60.0	40.0
19	0	-2	0	0	3.50	40.0	60.0	40.0
20	0	2	0	0	3.50	70.0	60.0	40.0
21	0	0	-2	0	3.50	55.0	20.0	40.0
22	0	0	2	0	3.50	55.0	100	40.0
23	0	0	0	-2	3.50	55.0	60.0	5.00
24	0	0	0	2	3.50	55.0	60.0	75.0
25	0	0	0	0	3.50	55.0	60.0	40.0
26	0	0	0	0	3.50	55.0	60.0	40.0
27	0	0	0	0	3.50	55.0	60.0	40.0
28	0	0	0	0	3.50	55.0	60.0	40.0

Metallographic Techniques

Specimen Preparation

Sample were prepared by cutting into pieces. These were grinded with successively finer grade of many papers from 120 to 1000 grit to produce a reasonably flat surface and polished using (0.5 μ m) alumina past. Then cleaned with acetone (10min.), ethanol (10min.), rinsed with distilled water (10min.) and dried. The specimens surface needs to be optically flat acting as a perfect mirror and free from any contaminates. The contaminates can cause failure of the coating by way of preventing abond from developing and also may contribute to the rusting of the steel substrate. Inadequate surface profile may result in the coating having lower bond strength and this may cause the coating to delaminate. Therefore, the polished specimens keep in disc cater with silica gel to prevent any scale.

A heat treatment annealing of stress relief before anodizing process in which the specimen is heated to the recommended temperature, the steel is heated to a temperature below or close to the lower critical temperature (550-650) $^{\circ}$ C, held at this temperature for some time and then cooled slowly [12]. held there long enough to attain a uniform temperature, and finally cooled to room temperature.

Anodizing Steps

A method of anodizing steel, including the steps of:

1. The specimen was weighed before anodizing (W_1)
2. Connecting a steel to a positive terminal of a power supply.
3. Connecting a counter electrode (stainless steel) to a negative terminal of the power supply.
4. Placing the steel and counter electrode into a solution of KOH in the vessel.
5. Applying a voltage across the terminals to anodize the steel by growing anodic oxide film directly on a steel surface of the object.
6. The solution is stirred and heated during step of applying a voltage.
7. Digital multimeter type (VICTOR 98A) interface is connected to the desktop computer and power supply.

After the requirements of experiment are set up, the power supply is switched on a fixed constant voltage at the desired value. The electrodes are immersed in the solution while the power supply is switched on, in which current density increased gradually. Before the power supply is switched off and at the end of the desired time, the anode specimen is removed from anodizing cell immediately to prevent the dissolution of anodic film, then it is rinsed with distilled water to remove the excess solution on the specimen. Later the specimen is dried by using drying furnace at 50 $^{\circ}$ C for 30 minutes and stayed in dissector about 30 minutes. The sealing process use when the anodizing process is completed, the specimen is dipped in a flask filled with hot distilled water and kept for 20 minutes at 97-99 $^{\circ}$ C to seal porous anodic film, and then specimens are dried in drying furnace at 50 $^{\circ}$ C for 30 minute. The specimen is weighed after anodizing (W_2). A schematic diagram of anodizing apparatus and the steps of processes before and after anodizing are shown in figures (1) and (2) respectively. The microhardness of the specimens before and after anodizing process was evaluated on a Digital microhardness tester using a Knoop indenter (HVS-1000, LARYEE, china, 2012).

Table (4) Standard and analytical chemical compositions of carbon steel alloy [13].

Metals	Standard by ASTM A568	Analysis by (Wt.%)
Iron	Rem.	98.8
Carbon	0.20	0.2
Manganese	0.60	0.62
Aluminum	-	0.03
Copper	0.20 min	0.23
Silicon	-	0.02
Sulfur	0.04	-
Phosphorus	0.04	-

Results and Discussion

The Box–Wilson statistical experimental design was employed to determine the effects of operating variables on thickness and to find the combination of variable conditions resulting in optimum thickness.

Coefficients of the response functions for model

The STATISTICA computer program was employed for the determination of the coefficients of equation (3) by regression analysis of the experimental data for each where; T is thickness, b_0 is constant, $b_1 b_2 b_3 b_4$ are linear coefficients, $b_{12} b_{13} b_{14} b_{23} b_{24} b_{34}$ are cross product coefficients and $b_{11} b_{22} b_{33} b_{44}$ are quadratic coefficients. The calculated coefficients are listed in table (5) for mild steel were used in calculating predicted values of thicknesses.

$$T = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 + b_{44}X_4^2 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{23}X_2X_3 + b_{24}X_2X_4 + b_{34}X_3X_4 \dots (3)$$

Consequently, an estimation of the mean model is found by substituting estimations for the coefficients, resulting in equations (4).

$$T = 2.949 + 0.781X_1 + 0.131X_2 + 1.240X_3 + 0.334X_4 + 0.047X_{11} + 0.003X_{22} + 0.377X_{33} - 0.153X_{44} + 0.041X_{12} + 0.115X_{13} + 0.026X_{14} + 0.046X_{23} + 0.025X_{24} + 0.141X_{34} \dots (4)$$

Then, the predicted thicknesses obtained from substitution in equation (4) are summarized in table (6).

Table (5) Coefficients of the response functions

Coefficients	Values of Coefficients
B_0	2.949
B_1	0.781
B_2	0.131
B_3	1.240
B_4	0.334
B_{11}	0.047
B_{22}	0.003
B_{33}	0.377
B_{44}	-0.153
B_{12}	0.041
B_{13}	0.115
B_{14}	0.026
B_{23}	0.046
B_{24}	0.025
B_{34}	0.141
Correlation Coefficient(R)	0.944729

The optimum conditions

According to Equations (3), using Hook and Jeeves pattern conditional method in terms of optimum thickness of the anodic film; the optimum conditions for anodizing carbon steel alloy are shown in table (7). The current versus time data for the optimum condition of specimen is shown in figure(3). With progress of time, the current density increases due to the dissolution reaction. Then, the current density reaches its maximum and then remains constant until the end of anodizing process because the stationary state of dissolution and film formation.

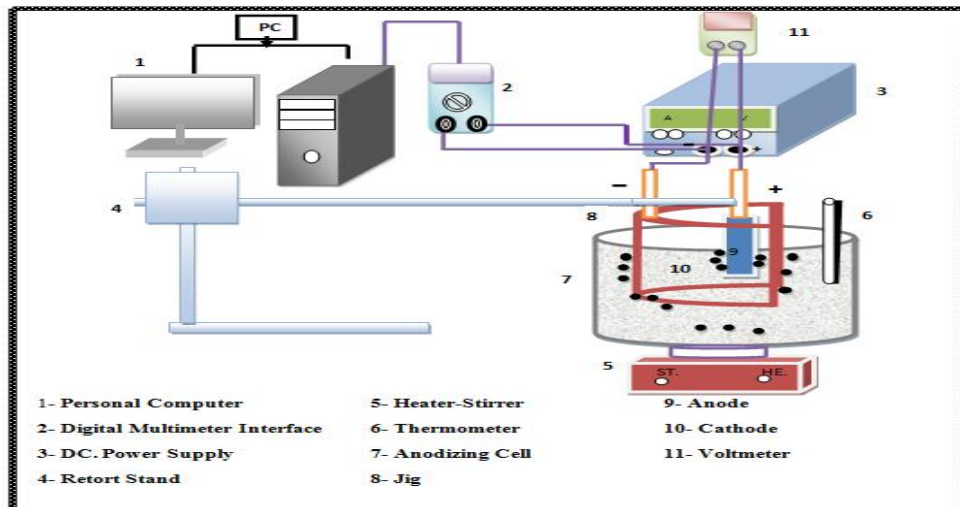


Figure (1) Schematic diagram for the whole assembly of the anodizing apparatus.

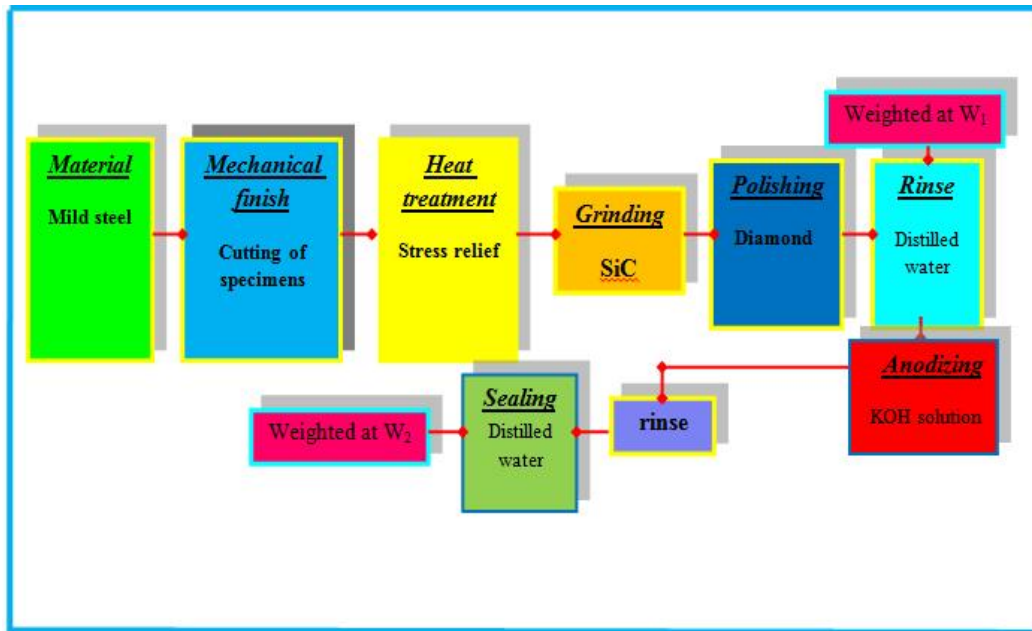


Figure (2) Typical process sequence for anodizing operations.

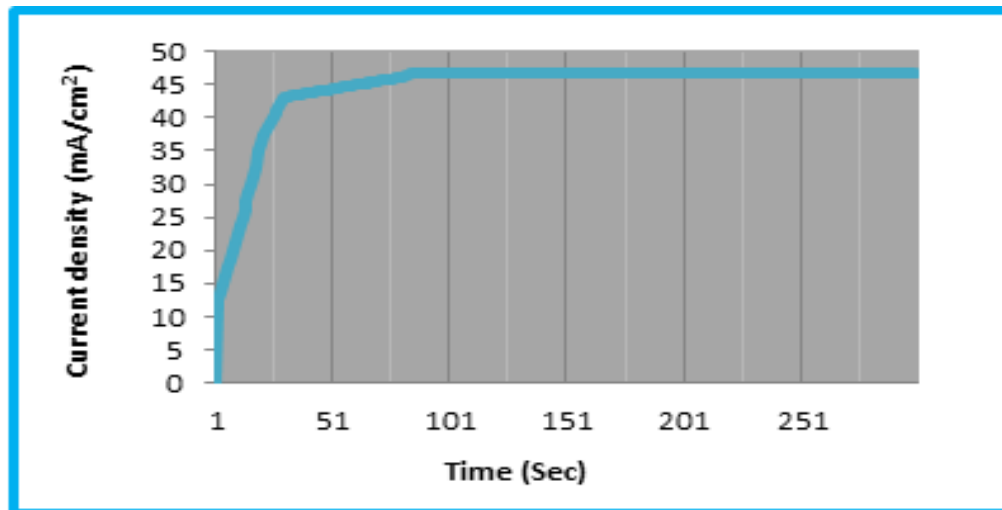


Figure (3) The Current-Time transient for mild steel at optimum condition after anodizing at constant potential

Table (6) Experimental thicknesses were compared with the predicted thicknesses for mild steel.

Exp. No.	Coded variables				Real variables				Exp. Thickness T (μm)	Predicted Thickness t(μm)	Corresponding Residual e _i =T-t(μm)
	X ₁	X ₂	X ₃	X ₄	Volt. (V)	Conc. (Vol. %)	Tem. (°C)	Time (Min.)			
1	-1	-1	-1	-1	2.25	47.5	40.0	22.5	1.600	1.523	0.951875
2	1	-1	-1	-1	4.75	47.5	40.0	22.5	2.620	2.535	0.967557
3	-1	1	-1	-1	2.25	62.5	40.0	22.5	2.000	1.609	0.804500
4	1	1	-1	-1	4.75	62.5	40.0	22.5	2.775	2.713	0.977658
5	-1	-1	1	-1	2.25	47.5	80.0	22.5	3.150	2.721	0.863810
6	1	-1	1	-1	4.75	47.5	80.0	22.5	4.000	4.317	1.079250
7	-1	1	1	-1	2.25	62.5	80.0	22.5	3.300	2.919	0.884545
8	1	1	1	-1	4.75	62.5	80.0	22.5	4.200	4.607	1.096905
9	-1	-1	-1	1	2.25	47.5	40.0	57.5	2.400	1.821	0.758750
10	1	-1	-1	1	4.75	47.5	40.0	57.5	2.800	2.937	1.048929
11	-1	1	-1	1	2.25	62.5	40.0	57.5	2.510	1.951	0.777291
12	1	1	-1	1	4.75	62.5	40.0	57.5	2.900	3.159	1.089310
13	-1	-1	1	1	2.25	47.5	80.0	57.5	3.800	3.619	0.952368
14	1	-1	1	1	4.75	47.5	80.0	57.5	5.100	5.319	1.042941
15	-1	1	1	1	2.25	62.5	80.0	57.5	3.950	3.861	0.977468
16	1	1	1	1	4.75	62.5	80.0	57.5	5.820	5.653	0.971306
17	-2	0	0	0	1.00	55.0	60.0	40.0	0.300	1.437	4.790000
18	2	0	0	0	6.00	55.0	60.0	40.0	4.960	4.241	0.855040
19	0	-2	0	0	3.50	40.0	60.0	40.0	2.720	2.853	1.048897
20	0	2	0	0	3.50	70.0	60.0	40.0	2.990	3.273	1.094649
21	0	0	-2	0	3.50	55.0	20.0	40.0	1.880	2.353	1.251596
22	0	0	2	0	3.50	55.0	100	40.0	6.100	6.045	0.990984
23	0	0	0	-2	3.50	55.0	60.0	5.00	1.900	2.043	1.075263
24	0	0	0	2	3.50	55.0	60.0	75.0	3.110	3.387	1.089068
25	0	0	0	0	3.50	55.0	60.0	40.0	2.930	2.963	1.011263
26	0	0	0	0	3.50	55.0	60.0	40.0	2.950	2.963	1.004407
27	0	0	0	0	3.50	55.0	60.0	40.0	2.980	2.963	0.994295
28	0	0	0	0	3.50	55.0	60.0	40.0	2.990	2.963	0.990970

Effect of optimum conditions on film thickness

The effect of anodizing mild steel at optimum conditions and different concentration is illustrated in figure (4).

Table (7) The optimum conditions of anodizing mild steel.

Conditions	Values of optimum conditions
Voltage (V)	1
Concentration (Vol. %)	40
Temperature (°C)	20
Time (min)	5

It can be shown that at low concentration of (40%), the film thickness increases and at higher concentration of (70%), the film thickness decreases due to increase the film dissolution rate.

Figure (5) shows, the effect of different voltages on thickness for mild steel. An increase in voltage leads to increase in thickness of anodic oxide film due to increase in growth rate.

In figure (6), it can be shown that an increase in thickness of anodic oxide film of mild steel is observed with an increase in temperature.

An increase in anodizing time leads to decrease in film thickness for mild steel as shown in figure (7). This is due to that longer time for anodizing at optimum conditions increases film dissolution reaction.

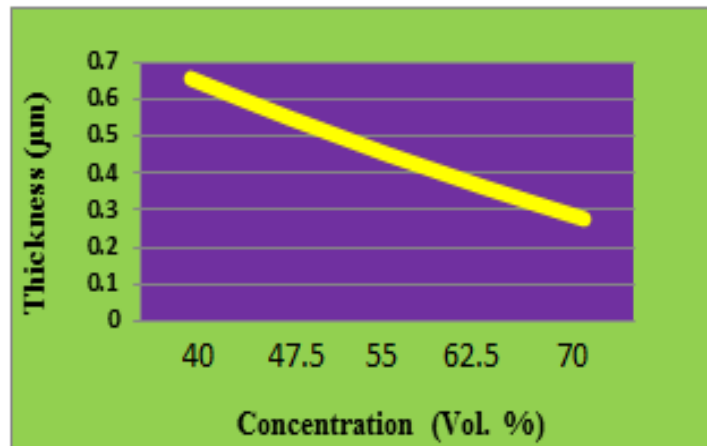


Figure (4) Effect of different concentrations on film thickness at optimum condition, for anodizing mild steel.

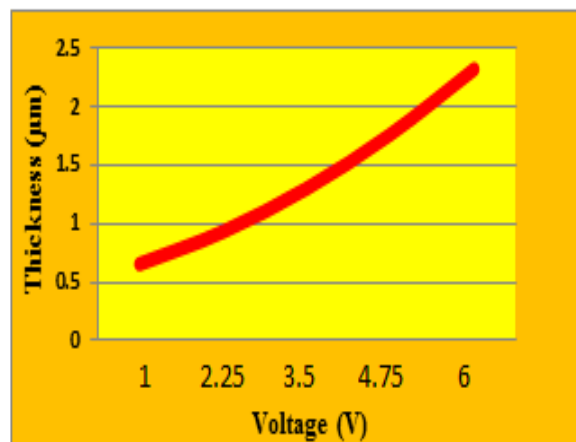


Figure (5) Effect of different voltages on film thickness at optimum condition, for anodizing mild steel.

T. D. Burleigh, et al. illustrated that higher voltages, higher temperatures, and higher concentrations lead to increase the rate of growth of anodic film thickness [14].

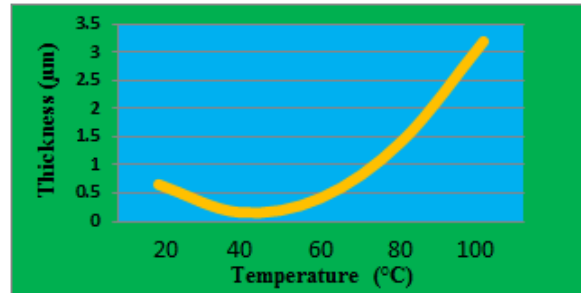


Figure (6) Effect of different temperature on film thickness at optimum condition, for anodizing mild steel.

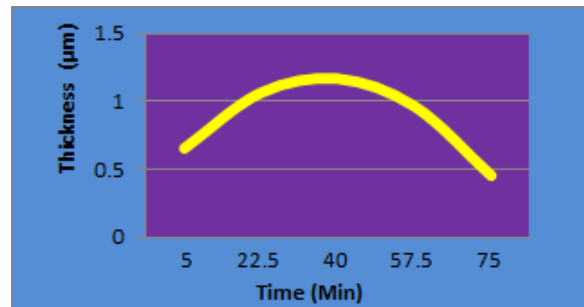


Figure (7) Effect of different times on film thickness at optimum condition, for anodizing mild steel.

Figure (8) illustrates the cross-sectional SEM micrographs of anodized mild steel. The thickness of the anodic oxide layer on mild steel is [0.65µm] due to presence of (Mn) and (C) in the mild steel .

Voon, et al. investigated that when the Mn content increases, the current efficiencies increase, and reduces the uniformity of the structure and this is contradictive to the reduced thickness and current density with the Mn content [15].

Anodic oxide films with different colors are observed on mild steel anodized at different anodizing conditions indicating in table (3), and shown in figure (9). Figure (9) shows different colors of anodic oxide film observed at different conditions. This can be explained due to the different oxide types present in the film observed by X-ray diffraction as shown in figure (10).

The images of the anodic film obtained for mild steel is shown in figure (11). From AFM images shown in the present, it can be concluded, after anodizing process, the average roughness of mild steel was [0.496 nm]. The pore diameter for mild steel was measured of [91.78 nm]. The average microhardness value is about (1083.5)Hk before anodizing process, and average microhardness value of anodized mild steel will be about (1477.5)Hk. Microstructure of mild steel with and without anodizing are observed in

figure (12). Typical microstructure of mild steel is observed in figure (12.A). Microstructure of anodized mild steel at (47.5%) KOH concentration and time of (22.5min) is shown in figure (12.B). It illustrates nonuniform distribution of anodic oxide film through the grains. Microstructure of mild steel at (62.5%) KOH concentration and time of (57.5min.) is also observed in figure (12.C). It indicates the uniform distribution.

It was initially thought that this film varied in thickness from grain to grain, according to their crystallographic orientation, and that the birefringent properties of the oxide film varied the ellipticity produced by reflection of the beam. Instead, the coloration effects in polarized light are due to double reflection from a furrowed surface produced by the anodizing solution, similar to certain chemical etchants discussed previously [16].

CONCLUSIONS

The following conclusions are drawn from the results of the investigation:

- 1- The Box–Wilson statistical experimental design was employed to determine the effects of operating variables on thickness and to find the combination of variables resulting in optimum thickness. Using Hook and Jeeves method.
- 2- The optimum conditions for anodizing mild steel alloy are (1V, 40Vol. %, 20°C, and 5min). In the range of anodizing conditions, the thickness of anodic oxide film is increased and can be seen that in the order by
Temperature > voltage > time > concentration.
- 3- The thickness of anodic film of mild steel is different due to different anodizing conditions. The thickness of anodic oxide layer on mild steel was [0.65µm].
- 4- The AFM technique measures the pore diameter of the anodic film of mild steel which is [91.78 nm] and the roughness is (0.496nm).
- 5- The colors of anodic oxide film are formed on mild steel alloy and change with different conditions due to different oxide types present.
- 6- The average microhardness value is about (1083.5)Hk before anodizing process, and average microhardness value of anodized mild steel will be about (1477.5)Hk.

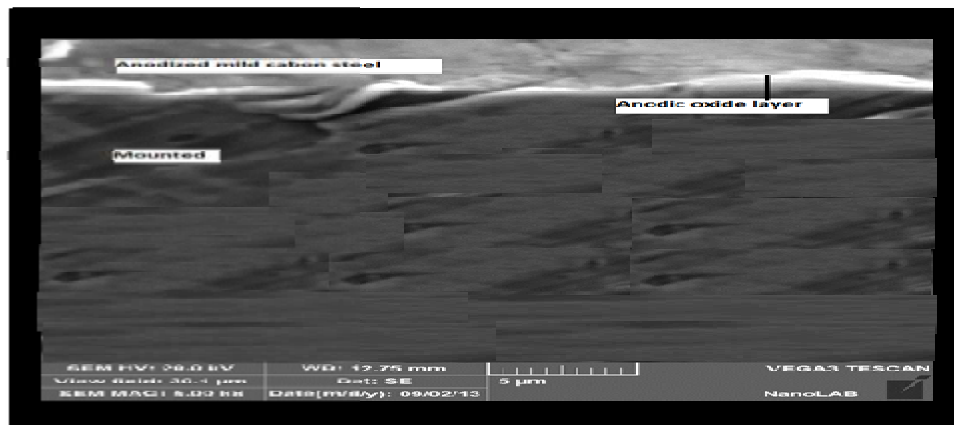


Figure (8) Cross-sectional SEM images of the anodic films formed at optimum conditions for mild steel

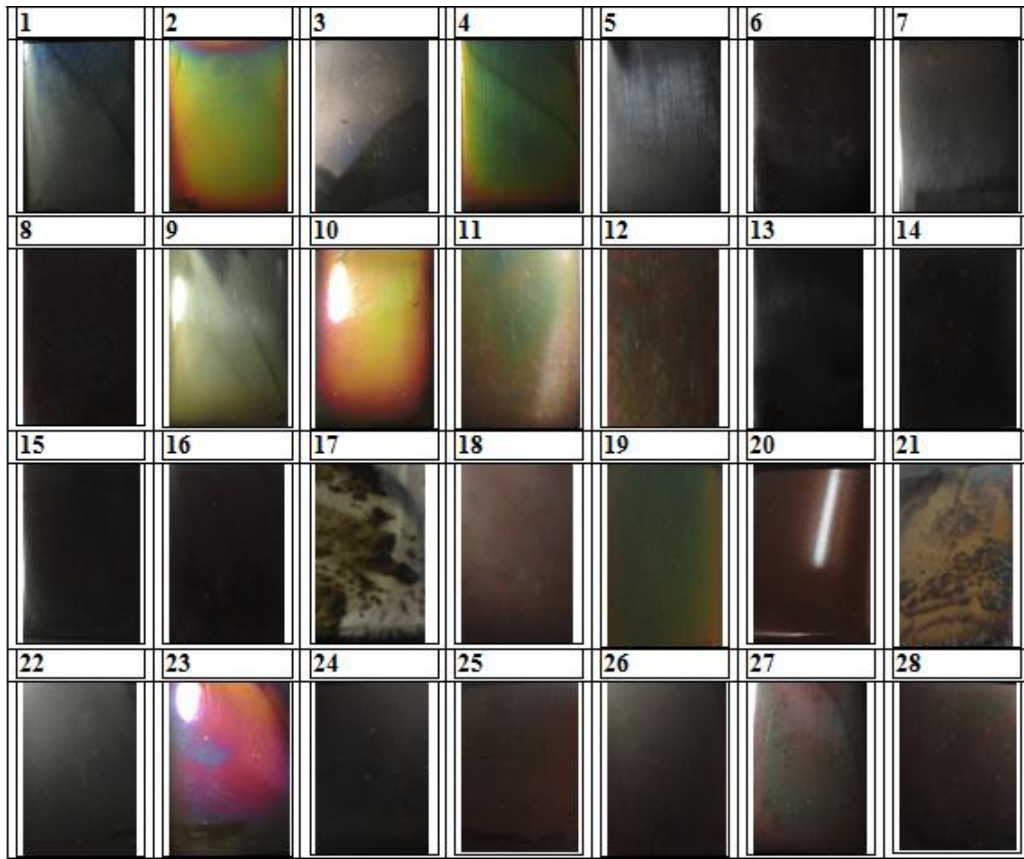


Figure (9) Different colors of anodic oxide films observed at different anodizing conditions.

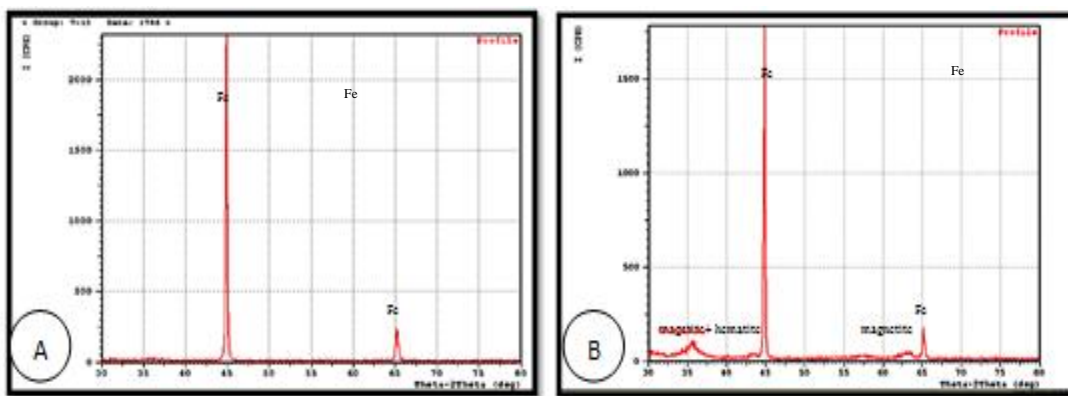


Figure (10) [A] XRD for mild steel after anodizing process at optimum conditions [B] XRD of anodizing mild steel at (100°C).

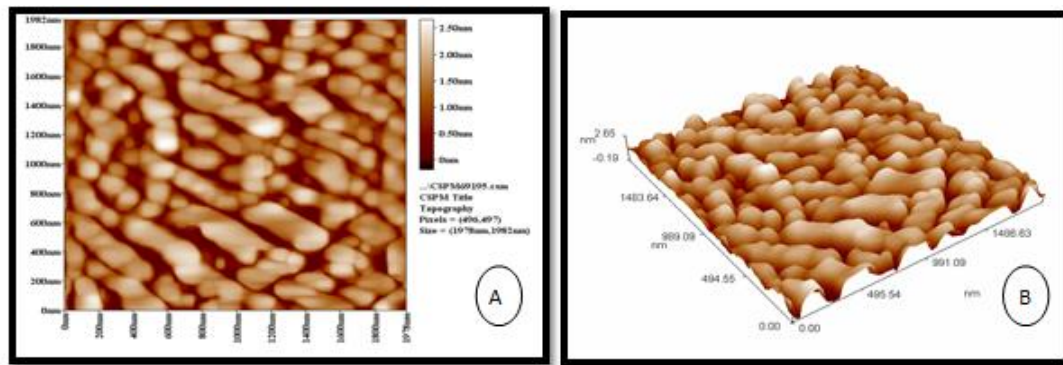


Figure (11) Simultaneously acquired AFM contact mode: (A) 2D-topography images, (B) 3D-topography images of the anodic oxide film for mild steel obtained at optimum conditions.

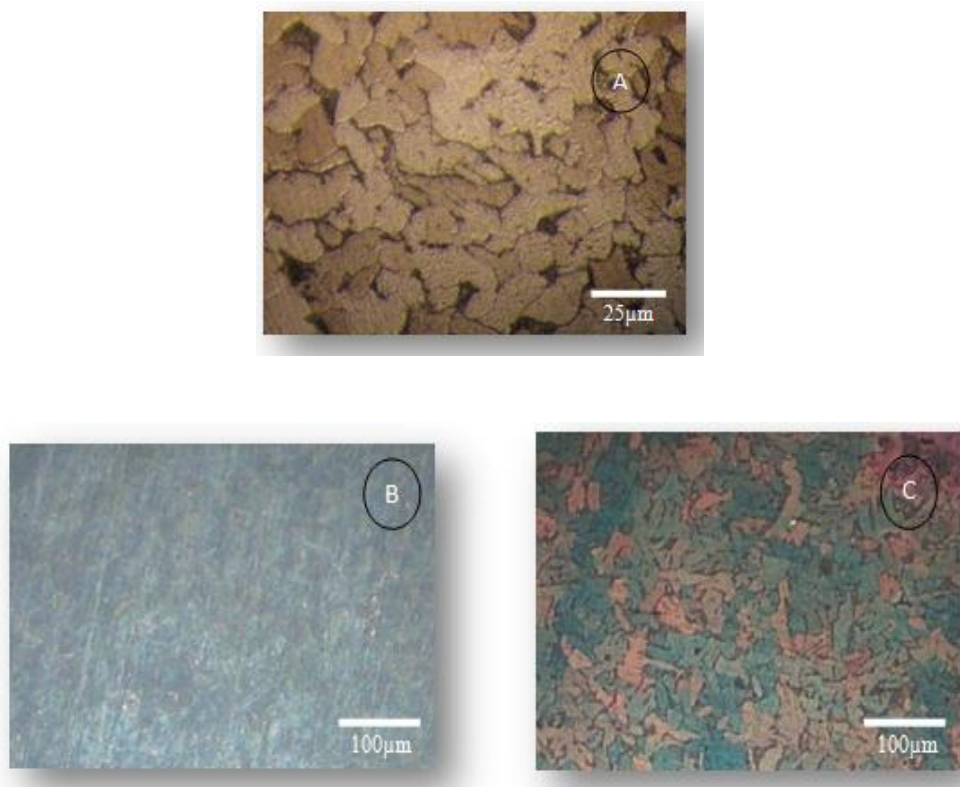


Figure (12) [A] Microstructure of cross section specimen before anodizing, [B] Mild steel anodized at (22.5min) and (47.5%), [C] mild steel anodized at (57.5min) and (62.5%).

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