OPTIMIZATION OF THE PROCESS PARAMETERS IN

ANODIZATION OF ALUMINUIM

By

ENYA R.   CHINYERE

(97/5992EH)

A research project submitted to the Department of Chemical Engineering, School of Engineering and Engineering Technology, Federal University of Technology, Minna, Niger State.

In partial fulfillment of the requirement for the award of Bachelor of Engineering (B.ENG) Degree in chemical Engineering

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DECLARATION

I hereby declare that this project work is my original handwork and has not being found written or done elsewhere.

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ENYA R. CHINYERE

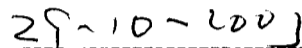.................................
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DEDICATION

This project work is dedicated to God Almighty, perfection of Beauty who shined his light and favor on me. Also to my lovely parents, sisters and brothers.

CERTIFICATION

This is to certify that this research project titled "Optimization of the process parameters in Anodizattion of Aluminium was carried out by ENYA R. CHINYERE under the supervision of Dr.Aloko Duncan and submitted to the Chemical Engineering Department, Federal University of Technology, Minna in partial fulfillment of the requirements of the award of Bachelor of Engineering (B.ENG) degree in Chemical Engineering

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Dr. Aloko Duncan   **Signature and Date**

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Dr. F. O. ABERUAGBA   **Signature and Date**
Head of Department

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External Examiner   Signature and Date

ACKNOWLEGDEMENT

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ABSTRACT

The optimization of the process parameters in anodization of aluminium was carried out using factorial design of the first order.

The relationship between thickness of anodization ($\hat{U}$), with voltage (X_1) and time of anodizing (X_2) is given as

$$\hat{U} = 32.074 + 10.189X_1 + 2.156X_2$$

The individual effect of voltage and time on the thickness of anodized aluminium are 10.189 and 2.156 respectively while their joint effect is insignificant.

The response surface graph of the equation was plane, therefore the optimum operating conditions could not be found.

CHAPTER ONE

1.0 INTRODUCTION

1.1 BACKGROUND

The surface of aluminium in its normal state in contact with air have good shine and corrosion resistant properties due to an oxide film formed on its surface, which unable to provide reliable protection against corrosion. The thickness of the oxide film can be increased artificially by electrochemical methods. This process may be called anodic oxidation or anodizing. As clear from the word anodizing, in a proper electrolyte anode is provided of pure aluminium. Anodising is the common designation for the anodic oxidation of certain metals to form stable oxide films on their surfaces.(A.D.A,1989)

Anodic oxide coatings are formed on aluminium and its alloy in a variety of media. Anodic oxidation is performed to produce decorative finishes, for architectural purpose or to increase the corrosion or abrasion resistance of the substrate metal. The most important anodisation process is that for aluminium and consequently the research is devoted to aluminium and its alloys.

The coating thickness has a direct correlation to corrosion resistance with test indicating that the coating thickness is the most important single factor in corrosion resistance of anodic films (Charles&Raymond,19992).Operating parameters such as voltage, time of anodizing, electrolyte temperature among many others are of importance in the formation of a more stable film. In past years anodisation has been carried out under different operating conditions, producing films of varying thickness which might be a highly corrosion resistance metal or otherwise. From the many use of aluminium, a high corrosion resistance metal is needed to meet the high demand of aluminium and the question is at what operating conditions do we obtain an optimum thickness, hence a high corrosion resistant metal. In answering the above question, a

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series of experiments need to be performed at different levels to actually observe the effect of these factors and their interaction. This procedure has been found to be time consuming, tedious and also expensive. Therefore, the need for better techniques in which the desired information can be obtained from a small amount of data thus the factorial design experiment becomes handy. Many of the early application of experimental design methods were in the agricultural and biological sciences. However, the first industrial applications of experimental design began to appear in the 1930s, initially in the British textile and woolen industries (Douglas, 1991).After world war 11, experimental designs methods were introduced to the chemical and process industries in the United States and Western Europe

In recent years, there has been a revival of interest in experimental design in United States because many industries discovered that their competitive success. The day is approaching (hopefully rapidly) when all engineers will receive formal training in experimental design as part of their undergraduate education. The successful integration of experimental design into engineering profession is a key factor in the future competitive of the industrial base of the United States. Much of the research in engineering science and industry is empirical and makes extensive use of experimentation. Statistical methods can greatly increase the efficiency of these experiments and often strengthens the conclusion so obtained (Douglas, 1991).

1.2 APPROACH

- A 2^2 factorial design approach was used, two factors at two level.
- A response surface equation was obtained in the form:

$$Y = b_0 + b_1x_1 + b_2x_2 + b_{12}x_{12} + ei$$

Where b_0 = mean effect of A and B

b_1 = main effect of A

b_2 = main effect of B

b_{12} = interaction effect of A and B

X_1 = coded sign in the A column of the designed matrix

X_2 = coded sign in the B column of the designed matrix

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X_{12} = coded sign in the AB column of the designed matrix

While A and B are voltage and time respectively.

- The adequacy of this equation was tested and a response surface graph drawn.

1.3 AIM AND OBJECTIVES

The aim is to determine the effectiveness of anodisation of aluminium as voltage and time were varied. While the objectives are to determine:

i) The effect of voltage and time on the anodisation of aluminium.

ii) Their interaction effect.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 DISCOVERY OF ALUMINIUM

Aluminium is now produced in such quantity that in terms of volume it ranks second to steel among the industrial metals.(A.D.A,1989).This position appears all the more remarkable when it is recalled that the metal itself has been known only about 130 years and indeed, the industrial history of aluminium did not begin until 1886,when Paul Heroult in France and Charles Martinhall in the U.S.A. independently discovered that it could be produced by passing an electric current through a bath of molten cryolite in which aluminium oxide was dissolved. The discovery in 1886 of the electrolyte process for the commercial production of aluminium led to the development of one of the most important engineering materials. The importance of aluminium is due primarily to a combination low density and high strength.

In the very early days of the industry, there was little demand for the pure metal, but as its unique combination of properties and the possibility of varying them by alloying with other metals become known, the demand increased, production rose and the price fell. The growing importance of air transport has been a main cause of the rise of the aluminium industry and it may be remarked, the growth of air transport has itself been made possible by the achievements of
the industry in producing light alloys of improved quality and strength. Today aluminium of commercial purity contains 99.8%of pure metal. A further refining process makes it possible to produce aluminium of supper purity(99.99%) and this is used for certain special application, such as lighting reflectors(A.D.A,1989).

Aluminium really began to achieve industrial importance in the period 1900-1914, the chief contribution being the discovery of age-hardening by the German Chemist Wilm

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in 1907. He had been trying to harden an aluminium alloy containing copper and magnesium by heating it to 500 degree Celsius and quenching in water, the experiment was unsuccessful-the alloy was still soft. A little later Wilm found that without further treatment it became harder and on further investigation he discovered that the strength had increased and considerably while the alloy had been lying for few days at room temperature following the quench. This chance discovery was confirmed, thus opening the way for the development of aluminium alloys as structural material with strength equal to that of steel (A.D.A, 1989). Wilms discovery was the basis of the first Duralumin patent, taken out in 1909. At about the same time a research team at Royal Arsenal, Wool Wich, working independently but on other alloys, made a similar discovery which, however was not developed commercially.

In 1911 a modest beginning was made in Birmingham of the commercial development of the first high- strength alloy based on Wilm 's patent .Before 1914 ,the material was being produced in the form of sheet, tube and extruded sections and was used in constructing the early rigid airships. The production of aluminium has risen with the needs of the engineer in field after field application.

2.2 SOURCES OF ALUMI NIUM

Aluminium is the most abundant metal in the earth crust but it is always combined with other elements such as oxygen, silicon, sodium etc. (A.D.A, 1989). Aluminium is an important constituent of nearly all common rocks except sandstone and limestone, but the only commercial source of the metal is bauxite.

2.3 PROPERTIES OF ALUMINIUM

The surface of pure aluminium is covered with a thin inert film of oxide which renders the metal practically immune to attack by the atmosphere and by many chemical substances such as acid, ammonia and sulphuretted hydrogen.Certain substances notably caustic soda and other alkalis and some acid dissolves the oxide film so that the metal is no longer protected. The workharding alloy has approximately the same resistance as the pure metal is no longer protected. The

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workhardening alloy has approximately the same resistance as the pure metal. Copper as an alloying element reduces the durability of the durability of the metal, but by comparison with iron and steel the resistance, the resistance to most form of attack is high for all the alloy. Aluminium alloys neither rust nor cause the staining of other materials in contact with them. The immunity of aluminium and many of its alloys to chemical action is one of the main reasons for its wide use. Aluminium alloys are light in weight with comparatively high strength. It also has high electrical conductivity and ductility.

2.4 ECONOMIC PROSPECT OF ANODISATION PROCESS

The consumption of anodized aluminium in the world today cannot be over emphasized. The use of the material finds its way virtually in all manner of technological advancement. To mention but a few is the use of anodized aluminium in the following areas;

   i. Aluminium furniture as in the industries, home and offices.

   ii. In the automobile: the use of aluminium car manufacturing rising at an almost incredible rate

   iii. Cotton rails etc

Confirming the positive trend, the economy of it is that;

   i. Many aluminium producers are expanding their plants thereby creating employment for larger number of people.

   ii. At personal level it creates market for many individual.

   iii. The anodizing materials are highly enhanced since this increases the quality of architectural works e.g. in construction of sliding doors and windows, in construction of aircraft and ships.

2.5 MECHANISM OF ANODISING AND STRUCTURE OF THE ANODIC FILM

Anodic oxide coatings are formed on aluminium and its alloys in a variety of media. The principal (commercially used) electrolytes are solution of sulphuric acid , chromic acid , oxalic acid and boric acid, although many other electrolyte have been studied as anodizing media, for example phosphates, phenols and organic acids.

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The anodic film is aluminium oxide, i.e. Al_2O_3 and has a dense structure in that there is a thin non porous inner oxide adjacent to the metal, the barrier layer, and a thick porous outer oxide (fig 2.1).The outer porous oxide has a cellular structure, the cell size being determined by the electrolyte temperature and composition, however, is determined by the anodisation voltage. The overall thickness of the oxide is largely determined by the coulombs passed in the oxidation process. The pore diameter and cell size are dependent on both the electrolyte and the formation voltage (frounttofer, 1976). The outer porous film is composed of partially hydrated alumina whilst the compact inner layer is alumina. The outer heavy layer of the anodic coating is assumed to be more porous with the pores stacked somewhat like parallel tubes extending through the layer from the surface heavily down to the barrier layer. The anodic layer is always being formed from the underlying aluminium surface and the previously termed oxide. The oxygen ion from the electrolyte, under the influence of the impressed voltage, travels through the pore channels to reach the aluminium-ion-rich area at or near the base of the barrier layer.

The major conflict between theories concerns the reaction of the barrier layer. Whether this layer has a rectification effect and allows passage of the aluminium ion outward only, whether the barrier layer breaks down at weak point as the voltage is raised or whether the layer is constantly breaking up and returning, the sequence is not generally agreed upon. As the mechanism on the oxygen ion traveling through the pore structure towards the barrier continues, the scribbling action on the pores near the outer part of the layer tends to develop a funneling of the pore channels which then becomes larger near the surface and taper smaller inward. This tapering effect is also accelerated by the concurrent dissolution action of the electrolyte dissolving the surface of the oxide layer and reducing thickness of the pore walls near the surface. As the thickness of the anodic increases, the impressed voltage needed to sustain the action increases. The final thickness is determined by the terminal voltage, temperature, solution, time and other operating variables (Charles and Raymond, 1992).

The mechanism of anodic oxidation of aluminium is still not fully established but the following is generally accepted. When the metal is anodized at 10-25V in a suitable

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electrolyte, there is a strong electric field across the thin air-formed or natural oxide film. As a result of this high field, there is an appreciable current density Al3+ ions migrate from the aluminium through the air-formed film into the anolyte and react with hydroxyl ion or oxygen produced at the anode from the electrolyte. Aluminium oxide is formed at the oxide-anolyte interface and as the surface oxide increases in thickness, the current density decreases and surface dissolution of the oxide by the electrolyte creates porosity in the film. Current flow then increases again and there is internal oxidation of the aluminium such that the film grows with progressive oxidation of the aluminium, the inner region of the oxide remains compact (the barrier layer) whilst the outer region is porous. The total thickness of the metal and oxide is virtually equal to that of the original metal; although there is some net loss of aluminium (Fraunttofer, 1976).The barrier layer adjacent to the metal achieves a relatively constant film thickness whilst the outer porous oxide grows with continued anodic oxidation.

The barrier film thickness is determined by the applied voltage, being in of the order of 0.015-0.020um/v (Fraunttofer, 1976).Out ward diffusion of Al_3^+ ions inward diffusion of O2- ions occurs across the barrier film. The outer film forms by breakdown of the inner film and there is simultaneous chemical attack of the outermost surface of the oxide by the outer most surface of the oxide by the electrolyte and formation of the inner oxide.

2.6 ANODISING KINETICS

There is strong evidence that the rate –controlling process for value metal anodisation is the migration of ions within the oxide film. It has been shown that the relation between the field intensity and the ionic current is not influenced by the film left on the metal after electrolytic polishing nor by different films produced by subsequent anodisation Guntherschule and Betz used an empherical equation to relate anodisation current to field strength

$$J=\alpha e^{\beta E}$$

Where α and β are constants, E is the field strength and j is the current density. Thus, it is assumed that ionic conductivity in the metal oxide is rate – controlling step in anodisation.

A plot of lnj versus E should be linear.
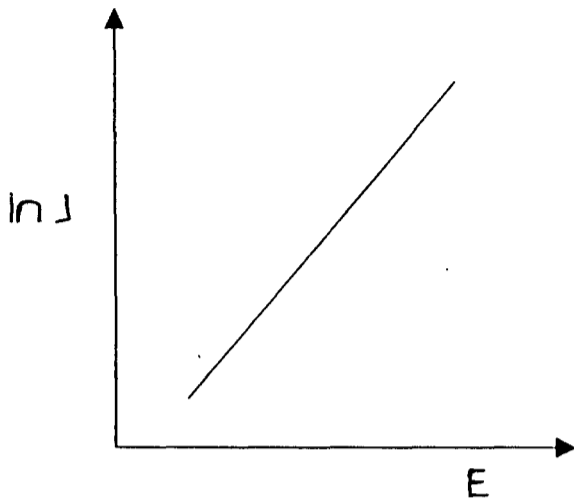
2.7 PREPARATION OF ALUMINIUM FOR ANODISING

Successful anodizing depends upon the pretreatment of the metal, the choice of the electrochemical process and the aluminium alloy being oxidized.

There must be no grease on the metal surface and the metal should be free from blemishes such as scratches, blow holes and pits since anodizing emphasizes such defects. The precise pretreatment schedule depends upon the final application of the anodized metal but generally the first treatment is abrasive, blasting scratch brushing or mechanical polishing. This is followed by degreasing and cleaning. An etching stage may be incorporated.

2.8 ANODISING ELECTROLYTE

The most commonly used electrolytes for anodizing aluminium are sulphuric acid and chromic acid separately and sometimes in combination Oxalic acid anodizing was developed in Japan and is used to some extent in Germany, although addition of oxalic acid (up to 5%) are sometimes made to sulphuric acid solutions for high operating temperature solutions or to increase the hardness of the anodic film,

particularly if lowered operating temperatures are impracticable. Anodic films formed in sulphuric-oxalic acid baths are generally more abrasion resistant than those formed in sulphuric acid alone.

The properties of the anodic film are determined by the anodizing condition, notably acid concentration, electrolyte temperatures and applied voltage (which determines the current density) (Fraunttofer, 1976). Harder films are produce at lower temperatures and in more dilute solutions whilst increase bath temperatures and higher voltage lead to more porous films (table 2.1).

The anodic film formed in chromic acid differs from that produced in sulphuric acid (table 2.2).

2.9 SEALING OF ANODIC FILMS

The anodic film on anodized aluminium consists of a thin layer and a thicker porous outer layer. The barrier layer dose not requires sealing but the outer layer must be sealed. The mechanism of sealing is not fully understood but is thought to involve conversion of the amorphous oxide of the pores into alpha-aluminamonohydrate, this conversion being accompanied by a change in volume. The volume change seals the oxide film by 'plugging' the pores so that anodic film becomes impermeable and its protective capability for the substrate metal is increased.

2.10 TESTING OF ANODISED COATING

Various test for the properties of anodic film on aluminium are cited in National and Military specification and these include test for thickness, continuity, sealing, staining and abrasion. Corrosion test are also undertaken and recently electrochemical tests involving impedance measurements and polarization procedures have been devised.

Coating thickness can be determined by dissolution of the anodic film from a known area of metal using chromic-phosphoric acid mixture, by microsectioning, using eddy current methods or by back-scattered electron techniques.

Pore

Porous
oxide

Borrier film
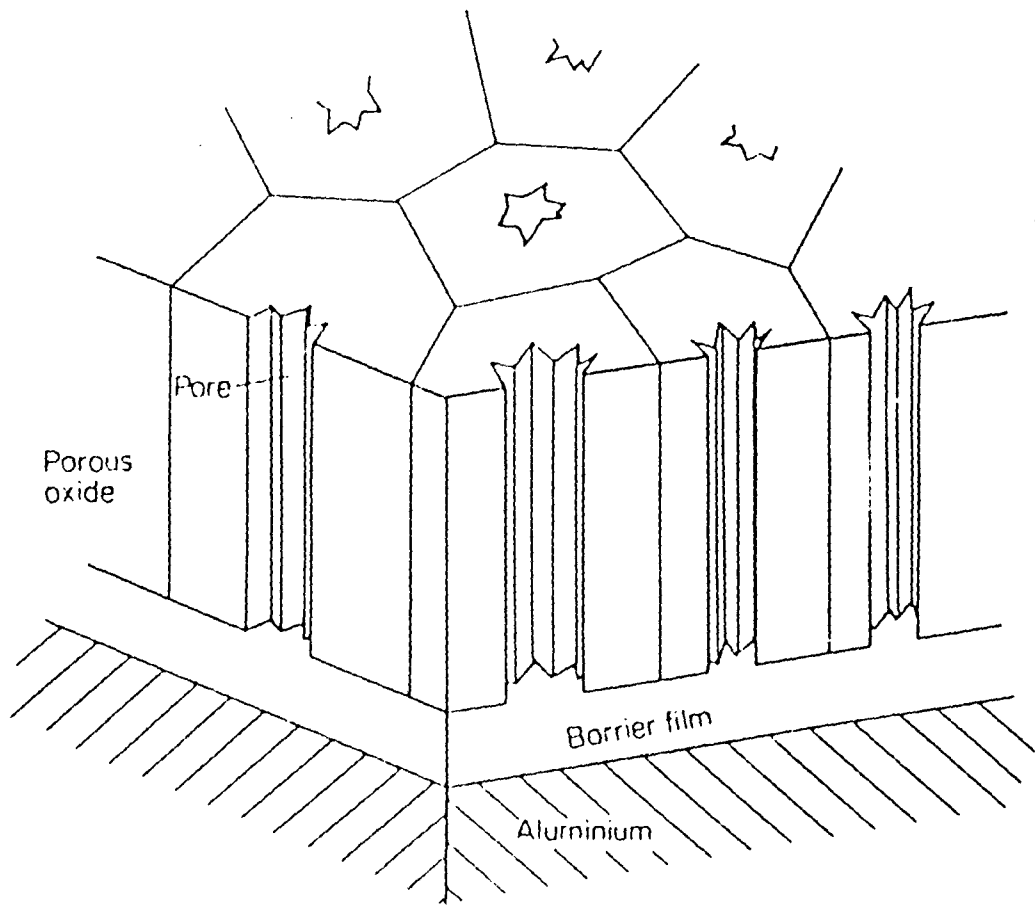
Aluminium

FIG. 2.1 SCHMATIC DIAGRAM OF ANODIC FILM FORMED ON ALUMINIUM

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TABLE 2.1 TYPICAL ANODISING OPERATING PARAMETERS FOR VARIOUS ELECTROLYTE

ELECTROLYTE	TEMPERATURE °F (°C)	OPERATING CONDITIONS		
		VOLTAGE	CURRENT DENSITIES $A/ft^2(A/m^2)$	TIME(min
Sulphuric acid (10-20% by weight)	60-80 (15-25)	10-25[a]	10-15 (10/6-161.5)[a]	20-10[b]
Chromic acid	95+/3.6 (35+/-2)	[c]	[c]	[c]
Hard coatings	25-50 (4-10)	[d]	20-25 (215.3-269)	60-200[e]

a; Depending on electrolyte concentration and temperature.

b; Longer anodizing time produce thicker films.

c ;During the first 10 minutes the potential is increased from 0 to 40volts. When the potential reaches 10volts, anodizing is continued for 20 minutes. At this potential, the current density will rang between $3.5A/ft^2$ (32.3-53 $8A/m^2$).After 20mins, the potential is increased from 40-50volts and then held for 5mins.

d; To maintain this current density the potential rises from 20-25v to40-60v.

e ;Thickness is approximately a linear function of time.

TABLE 2.2 COMPARISON OF THE ANODIC FILM PRODUCED ON ALUMINIUM IN SULPHURIC AND CHROMIC ACID

PROPERTY	SULPHURIC ACID	CHROMIC ACID
1. Opacity	Transparent	Translucent to opaque
2. Colour	Colourless, metallic luster.	Light grey
3. Dye effect	Clear bright colour with retained metallic lustre	Pastel and deep opaque colours
4. Average pore size (um)	1.2	2.4
5. Maximum film thickness (um)	50-100	25-50
6. Ductility	Moderate	High
7. Hardness	High	Moderate
8. Abrasion resistance	High	Moderate
9. Corrosion resistance	Good	Very good
10. Anodic process	Simple	Complex
11. Effect on alloys	High tolerance	Cannot be used for high copper or sillicon alloy

2.11 ANODISING APPLICATION

Anodizing is used for many purposes, usually with the application taking advantage of one or more of the specific properties of the anodic coating. The corrosion resistance of a painted part is largely derived from the paint system; however, paint adheres poorly to bare aluminium and the tightly adherent film acts as an intermediary for accepting the paint and binding it to the parts. The anodic layer also provides a portion of the overall corrosion resisting properties of the system. In aircraft applications, parts are usually chromic anodized, with the exterior given a primer and several top coats.

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One limited use of chromic anodizing is based on the extra ordinary penetrating ability of the chromic acid. The party to be inspected is anodized and lightly surface washed, then it is allowed to set for a period of time. If a crack is present the chromic acid may bleed out of the crack and appear as yellowish stain against the gray chromic anodized background.

Commercially, sulfuric anodized and dyed parts are used in automotive trim, cosmetic cases, aerosol caps, keys, picture frames, name plates and sporting goods among other applications. The integral color anodizing and variation of this process amounts for a large use of aluminium in out door architectural and building ap[placation.Hard coating is increasing in usage mainly because of its sliding wear and corrosion resistant properties. Also, like most hard coating surfacing, hard coating reduces fatigue strength up to 50%, depending upon the alloy, thickness of coating and the stress level involved. In aircraft application, where fatigue stresses are a design consideration the hard coat is kept out of radii and other notch sensitive areas.

Typical application for hard coating includes hydraulic cylinder bores, gun parts, and ordnance and missile components. Hard coating are also used in laser targets, paper mill rolls, fire hose connection, heat sinks, coarse screw threads, surveying instruments,aluminium gears, canes textile bobbins, pots and pans, sail boat mask,davits,and pulleys.

Anodic coating also have insulative properties. A break down voltage of 500 volts for a 0.001" to 0.002"(25 to 50μm) thick coating when measured ground to surface is normal expectancy. The increase in breakdown voltage is not directly linear with anodic coating thickness.

2.12 DESIGN CONSIDERATIONS

With the introduction of heavier anodic coatings, it becomes important to recognize the growth aspect involved in the formation of anodic coatings. As the aluminium oxide is being formed by dissolving aluminium from the surface of the parts and combining with oxygen from the bath, it both penetrates beyond the original surface and builds above the original surface. While the theoretical growth is approximately equal to one half of the total coating thickness. The growth portion of the coating is reduced by the solvent action of the electrolytes; the solvent action, in turn, is

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dependent upon the temperature, strength of the electrolyte, and exposure time in tank. In sulfuric anodizing the coating growth is approximately 30% of the total coating thickness and is a consideration on closely machined parts. In hard coating because of the refrigerated electrolyte, the coating growth approximately 50% of the coating thickness; in many application this becomes a design factor, especially on threads, dowell holes, close tolerance bores, and bearing diameters. A hard coating with a coating thickness of 0.002" (50μm) including building up and penetration of the coating would penetrate 0.001" (25μm) and build up 0.001" (25μm).

Another factor to consider in hard coating application is that the build up of the coating is perpendicular to the surface; at sharp edges, a corner effect results in a gap in the coating producing areas that are prone to chiping,especially as the coating thickness increases. If the corner is well radiused, the coating will approximate the curvature. A recommended radius for a 0.001" (25μm) thick coating is 1/32" (0.8mm); for a 0.03" (75μm) coating, 3/32" (2.4mm).

2.13 EQUIPMENT

The main components used in anodizing include tanks, racks, cathodes, agitators, ac power supply, cooling coils or heat exchangers and chillers, along with a ventilation system.

2.14 SAFETY AND ENVIRONMENTAL CONSIDERATIONS.

Anodizing processes on relatively safe because of the limited group and low concentrations of chemicals involved and the low closed circuit electrical requirements. Slipping on a wet platform is probably a more realistic danger than injury from the chemical involved. Chromic acid and bright dip operations are the most hazardous processes and requires proper ventilation. Handling any concentrated acid used for tank make ups requires proper safety clothing, goggles, gloves and safety toe boots. Only experienced personnel should handle the acid concentrates. Strong deoxidizers used in anodizing operations must be segregated to storage and handle properly. Because of the EPA federal laws all states are in the process of implementing control to ensure conformance. These controls, which are becoming more constructive and detailed, will ultimately result in isolation and sealing off the

anodizing plants to make it, in effect a leak proof container, sealed drains, leak proof floors with identification numbers. Copies of the manifest are forwarded to the state environmental office.

In general, chromates and phosphates used in anodizing present the major disposal concern. Evaporation systems operating under pressure to reduce evaporation temperatures and sedimentation.

2.15 FACTORIAL DESIGN AND APPLICATION

Factorial design are widely used in experiments involving several factors where it is necessary to study the joint effect of the factors on a response (Douglas,1991).

Factorial design is a statistical design which tests all combinations of effect all factors enabling:

A. All the main effect of every factor to be estimated independently of one another.

B. The determination of the dependency of the effect of any factor on the level of interactions.

C. The determination of all effects with maximum efficiency.

D. The estimation of the experimental error for the purpose of assessing the significance of the effect and enabling confidence limit be determined.

By statistical design of experiment, we refer to the process of planning the experiment so that appropriate data that can be analyzed by statistical method will be collected, resulting in valid and objective conclusions. The statistical approach to experimental design is necessary if we wish to draw meaningful conclusion from the data (Douglas, 1991).

The following terminologies are used in factorial design experiments:

FACTORS: A factor is a set of treatments which can be applied to experimental units. In doing this we have to be3 guided by economy and simplicity. Factors can be classified as qualitative and quantitative. The former includes those whose possible values cannot be3 arranged in order of magnitude. Conversely, a quantitative factor is one whose possible values can be arranged in order of magnitude and is usually associated with point on the numerical scale.

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LEVEL: A level of a factor is a particular treatment from the set of treatments which constitute the factor.

EXPERIMENTAL TREATMENT: An experimental treatment is the description of the way in which a particular unit is treated and comprises one level from each factor.

REPLICATION: This means a repetition of the basic experiment. Replication has two important properties. First, it allows the experimenter to obtain an estimate of the experimental error. This estimate of the experimental error becomes a basic unit of measurement for determining error. Secondly, if the sample mean (e.g.$\hat{U}$) is used to estimate the effect of a factor in the experiment, then replication permits the experimenter to obtain a more precise estimate of the effect.

2.16 THEORY OF FACTORIAL DESIGN

It can be shown that if the result of changing two or more factors is to be studied then in general the most efficient method is to use a factorial design method. Thus, efficient method is meant one, which obtains the required information with required degree of precision and minimum expenditure of effort (Davies, 1960).

In factorial design, statistical analysis such as an analysis of variance is used in detecting and separating the effect of factors, while regression analysis can be used to develop a quantitative relationship among the important factors. The approach of interpretation of data from factorial experiment consists of studying the relationship between the responses as dependent variate on the factor level. This approach helps to understand better how with the change in the level of application of a group of factors, the response change. A combination of the levels of the factors that leads to certain optimum response can also be located through this approach (Giri, 1979). Response surface methodology, or RSM, is a collection of mathematical and statistical techniques that are useful for the modeling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response. Suppose a process yield is a function of a level factor say factor X_1 and X_2, then

$$Y = f(X_1, X_2) + E$$

Where, E represents the noise or error observed in the response Y. If we denote the expected response by

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$E(Y) = f(X_1, X_2) = \dot{\eta},$

Then the surface represented by $\dot{\eta} = f(X_1, X_2)$ is called a response is called a response surface (Montgomery, 1991).

In most RSM problems, the form of the relationship between the response and the independent variables are unknown, thus, the first step in RSM is to find a suitable approximation for the true functional relationship between Y and the set of independent variable. Usually a low order polynomial in some region of the independent variable is employed .If the response is well modeled by a linear function of the independent variables, and then the approximating function is the first order model,

$$\hat{U} = b_0 + \sum_{i=1}^{k} b_i X_i + ei$$

The method of least squares is used to estimate the parameters in the approximating polynomial.The response surface analysis are then done in terms of the fitted surface. If the fitted surface is an adequate approximation of the true response function, then the analysis of the fitted surface will be approximately equivalent to analysis of the actual system. The model parameter can be estimated effectively if proper experimental design is used to collect the data. Design for fitting response surfaces are called response surface design (fig.4.1)

2.17 FACTORIAL DESIGN

The 2k design is particularly useful in the early stages of experimental work, where they are likely to be many factors to be investigated (Douglas, 1991). It provides the smallest no of runs with which k factors can be studied in a complete factorial design. Because there are only two levels for each factor, we must assume that the response is approximately linear over the range of the factor level chosen (Douglas, 1991). 2^2 factorial designs is the first design in the 2^k series with only two factors say X_1 and X_2 each run at two level. The levels of the factors may be arbitrary called "low" and "high". In the 2^2 design the low and high level of A and B are denoted by "-" and '+' respectively.

CHAPTER THREE

3.0 EXPERIMENTAL DESIGN METHOD

3.1 CONSTRUCTION OF DESIGN MATRIX TABLE

The design matrix table for the 2^2 full factorial which indicates the run by run experimental design, is shown in table 3.1. With two factors, two levels, a complete or orthogonalized design leads to a total of four experimental runs (Douglas, 1991). Let the applied voltage be factor A, and let the two levels of interest be 25Volts and 35Volts. The time of anodisation is factor B, with the high and low levels at 50 and 40 minutes respectively. The experiment replicated two times and the data are shown in table 3.2. The treatment combinations in this design are shown graphically in fig 3.1. By convention, we denote the effect of a factor by capital Latin letter. Thus "A" refers to the effect of factor A, "B" refers to the effect of factor B, and "AB" refers to the AB interaction. In the 2^2 design the low and high levels of A and B are denoted by "-" and "+" respectively, on the A and B axis. Thus,"-" on the A axis represents the low level of voltage (25Volts) whereas "+" represents the high level (35Volts), and "-" on the B axis represents the low level of time of anodisation whereas "+" denotes high level.

The four treatment combinations in the design are usually represented by lower case letters, as shown in fig 3.1. We see form fig 3.1, that the high level of any factor in the treatment combination is denoted by the corresponding lower case letter and that the low level of a factor in the treatment combination is denoted by the absence of the corresponding letter. Thus, a represent the treatment combination of A at the high level and B at the low level, b represent A at low level and B at high level, and ab represents both factors at the high level. By convention, (I) is used to denote both factors at low level. Therefore, the four treatment combination are summarized as follows (I), a, b, ab where,

(I) = factors A and B at low levels

a = factor A only at high level

b = factor B only at high level

ab = factors A and B at high levels

The design matrix table is constructed in the following way:

The columns are designated by the effect symbols (I), a, b, ab. Table 3.1 illustrates the arrangement for a two factor experiment. The sequence of "+" and "-" signs in the columns tells us how to combine the observation to obtain the main effect and their interactions .Column (I), having only plus signs, represents the average of the entire experiment. Column A, B are the main effects while column AB is obtained by multiplying the signs in the column headed by AB is obtained by multiplying the signs in the corresponding A and B columns.

TABLE 3.1 DESIGN MATRIX TABLE FOR 2^2 FULL FACTORIAL DESIGNS

TREATMENT COMBINATION	FACTORIAL EFFECT			
	I	A	B	AB
I	+	-	-	+
a	+	+	-	-
b	+	-	+	-
ab	+	+	+	+

TABLE 3.2 EXPERIMENTAL DATA

TREATMENT COMBIATION	REPLICATES		AVERAGE ($\hat{U}_u$)
	I Y_{u1}	II Y_{u2}	
A low, B low	19.55	19.82	
A high, B low	40.00	40.3	
A low, B high	23.67	24.5	
A high, B high	44.25	44.5	

FIGURE 3.1 TREATMENT COMBINATIONS IN THE 2^2 DESIGN
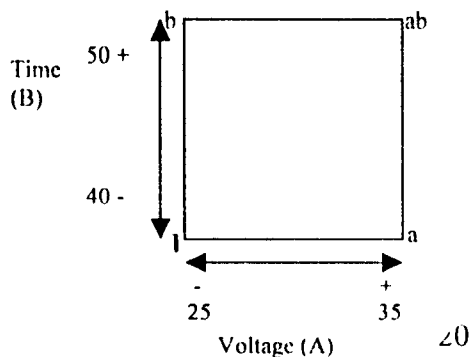
3.2 CALCULATING THE MEAN AND DISPERSION

Providing for the number of replicates, r =2, the mean value, $\hat{U}_u$ and the dispersion of the replicated observations, S^2_u in the u-th trial.

The mean of the replicated observation is given by:

$$\hat{U}_u = 1/r \sum_{u=1}^{r} Y_{uv} \qquad \text{--3.1}$$

Where r = number of replicates

$\qquad Y_{uv}$ = replicate observation

The dispersion of the replicated observation is given by

$$S^2_u = 1/(r-1) \sum_{u=1}^{r} (Y_{uv} - \hat{U}_u)^2 \qquad \text{---3.2}$$

3.3 CALCULATING G-VALUE

The homogeneity of the dispersion is determined using Cochron criteria.

$$G_{cal} = \frac{S^2_{u\ max}}{\sum_{u=1}^{N} S^2_u} \qquad \text{--3.3}$$

Where $S^2_{u\ max}$ is the max dispersion

$\sum_{u=1}^{N} S^2_u$ is the sum of dispersion

The G test is used to check if the factors of the replication have maximum accuracy of replication. It ascertains the possibility of carrying out regression analysis.

The condition of homogeneity is

$$G_{(\acute{a},\ (r-1),\ N)} > G_{cal} \text{---3.4}$$

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Where N is number of experimental runs

      r is number of replicates

      á is level of significance

3.4 CALCULATING MEAN SQUARED ERROR AND EXPERIMENTAL ERROR

The mean squared error is determined by

$$S^2_{(y)} = 1/N \sum_{u=1}^{N} S^2_u \quad \text{--3.5}$$

It is the average sample variance estimate.

The experimental error is given as

$$S_{(y)} = \sqrt{S^2_{(y)}} \quad \text{--3.6}$$

3.5 CALCULATING THE MEAN AND MAIN EFFECTS

Multivariate regression analysis was used for relating the variables. The functional relationship between the film thickness and the two factors Xi (i=1, 2) is formulated as a linear model:

$$\hat{U} = b_0 + b_1X_1 + b_2X_2 + b_{12}X_{12} + Ei \quad \text{---------------------------------------3.7}$$

Where the b's are regression coefficients of the model, the X's are the coded variables and Ei measures the discrepancy in the functional relationship and is a random error with zero mean and constant variance (Douglas, 1991)

The mean effect is given as

$$.b_0 = 1/N \sum_{u=1}^{N} (X_0\hat{U}_u) \quad \text{--3.8}$$

Where X_0 are the coded signs in the X_i columns of the design matrix.

The main effect is estimated thus

$$b_i = 1/N \sum_{u=1}^{N} (X_i\hat{U}_u) \quad \text{--3.9}$$

Where Xi are the coded signs in the A column of the design matrix.

The k- factor interactions is estimated by

$$b_{ij----k} = 1/N \sum_{u=1}^{N} (X_{ij----k} \hat{U}_u); \quad i \neq j \neq -------- \neq k \quad ----------------3.10$$

Where $X_{ij----k}$ are the coded signs in the A_{ij---k} of the design matrix table.

The quantities in brackets in equations 3.8, 3.9, and 3.10 are contrasts in the treatment combinations which is the total effect of each factor.

3.6 CONSTRUCTION OF CONFIDENCE INTERVAL AND TESTING OF HYPOTHESIS

Construction of confidence interval and testing of hypothesis about individual regression coefficients are frequently used in assessing their statistical significance. Confidence intervals for the regression coefficient, with confidence coefficient, á are of the general form.

$$b \pm t_{(á,N (r-1))} S_b --3.11$$

Where S_b is the estimated standard error in regression coefficient b and $t_{(á,N (r-1))}$ is an appropriate standard t- value with N(r-1) degree of freedom. For full factorial experiments, errors in each regression coefficient is the same and is determined by

$$S_{bo} = S_{bi} = S_{bij---k} = S_{(y)} \sqrt{N.r} --3.12$$

Where $S_{(y)}$ is the experimental error.

The statistical significance of the regression coefficient was tested by

$$t_{i---k} = \frac{\mid b_{i----k}}{\ldots\ldots\ldots \quad S_{bi----k}} --3.13$$

Where $\mid$ bi----k $\mid$ is the absolute value of the estimate of the coefficient being checked. The calculated t- values are compared with the appropriate critical value found from standard t- tables. A coefficient is considered significant if and only if

$$t_{cal} > t_{(á,N (r-1))} --3.14$$

For any coefficient that was statistically insignificant, such a coefficient will be left out of the regression model.

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3.8 CALCULATING THE SUM OF SQUARES

The adequacy of the model will be evaluated using null hypothesis ($H_o : b_{i=0}$) on the individual regression coefficients. The analysis of variance is very useful in confirming the significance of the coefficients (Douglas, 1991). In the 2^k factorial design with replicates, the regression sum of squares for any effect is

$$SS_R = r/N \text{ (contrast)}^2 \text{ ---}3.15$$

And has a single degree of freedom.

The total sum of squares will be calculated thus

$$SS_T = \sum_{u=1}^{N.r} Y^2_{uv} - \frac{\sum_{u=1}^{N.r} Y^2_{uv}}{N.r} \text{ --}3.16$$

The error sum of squares is given as

$$SS_E = SS_T - \sum SS_R \text{ ---}3.17$$

3.9 CALCULATING f – VALUES

Testing the significance of individual coefficient was carried out using fishers test (f-test)

$$f_{cal} = \frac{MS_R}{MS_E}$$

$$= \frac{SS_R/df_R}{SS_E/N(r-1)} \text{ --}3.18$$

Where df_R is degree of freedom in the regression.

The calculated f- values are compared with the appropriate critical table value. The null hypothesis will be rejected using:

$$f_{cal} > f_{[\acute{a},\, dfR,\, N(r-1)]} \text{ table}$$

With the conclusion that the coefficient contributes significantly to the regression (Douglass, 1991).

CHAPTER FOUR

4.0 RESULT AND ANALYSIS

4.1 FACTORS AND THEIR CODED LEVELS

Table 4.1 shows an estimation of upper and lower level for the two factors of voltage and time for the factorial design experiment.

TABLE 4.1: FACTORS AND THEIR CODED LEVELS

LEVEL OF FACTORS	CODE	INDEPENDENT VARIABLES	
		X_1 (Volts)	X_2 (mins)
Base level	0	30	45
Interval of variation	ΔX_i	5	5
High level	+1	35	50
Low level	-1	25	40

4.2 CALCULATION OF MEAN (YIELD) AND DISPERSION

TABLE 4.2 YIELDS FOR DIFFERENT TREATMENT COMBINATION

TREATMENT COMBINATION	REPLICATES		AVERAGE THICKNESS ($\hat{U}_u$)μm
	I Y_{u1}	II Y_{u2}	
A low, B low	19.55	19.82	19.685
A high, B low	40.00	40.3	40.150
A low, B high	23.67	24.5	24.085
A high, B high	44.25	44.5	44.375

4.3 COCHRAN TEST

$G_{(0.05, 1, 4) table} = 0.768$

$G_{(0.05, 1, 4) cal} = 0.753$

The adequacy of the data was tested. From the G-values, $G_{table} > G_{cal}$ which satisfies the condition for homogeneity hence the possibility of carrying out regression analysis. This also implies that the output factors of the replication have maximum accuracy of the replications.

4.4 CALCULATION OF EFFECT AND INTERACTION

The average effect of a factor defined as the change in response produced by a change in the level of that factor averaged over the levels of the other factor was calculated from equation 3.9.

TABLE 4.3 EFFECTS AND INTERACTION

TREATMENT COMBINATION	EFFECT
1	32.074
a	10.189
b	2.156
ab	-0.044

4.5 ANALYSIS OF VARIANCE

Analysis of variance is important in 2^k designs This enables us to examine the magnitude and direction of the factor effect and determine which variable are likely to be important .It is also important in assessing the statistical significance of the regression coefficient (bi)

4.5 ADEQUACY TEST

4.5.1 CALCULATION OF t-VALUES

TABLE 4.4 CALCULATED t –VALUE

TREATMENT COMBINATION	t_{cal}
1	268.346
a	85.245
b	18.040
ab	0.366

$t_{(0.05, 4)table} = 2.776$

t- TEST

A coefficient was considered significant if and only if $t_{cal} > t_{table}$. Table 4.5 shows which is significance or otherwise.

TABLE 4.5 THE ESTIMATED EFFECTS (b's) AND THEIR CALCULATED t-VALUES SHOWING THEIR SIGNIFICANCE

$b's$	t_{cal}
$b_0 = 32.074$	$t_0 = 268.346^a$
$b_1 = 10.189$	$t_1 = 85.245^a$
$b_2 = 2.156$	$t_2 = 18.040_a$
$b_{12} = -0.044$	$t_{12} = 0.366*$

a significant at 5%

* Insignificant at 5%

4.5.1.2 FISHER'S TEST (f-TEST)

Further test was carried on the regression coefficient in other to really affirm the significance of the obtained coefficient Using fisher's criteria a coefficient was considered a significance if

$$f_{(0.05, 1, 4)cal} > f_{(0.05, 1, 4)table}$$

4.6 f- TABLES

EFFECT	f-ratio
A	7265.84^a
B	325.42^a
AB	$0.134*$

a significant at 5 percent

Insignificant at 5 percent

$f_{(0.05, 1,4)\,table} = 225$

And when compared with the calculated f-ratios, it was found that compared that, Effect A and B are significant at 5 percent having met the condition for significance $f_{cal} > f_{table}$ while AB is insignificant at 5 percent.

TABLE 4.7 ANALYSISNG OF VARIANCE FOR REPLICATED

2^2 FULL FACTORIAL EXPERIMENTS

SOURCES OF VARIATION	EFFECT	SUM OF SQUARES (SS)	DEGREE OF FREEDOM (df)	MEAN SQUARES (SS)	f-RATIO
b_1	10.189	830.485	1	830.485	7265.84
b_2	2.156	37.195	1	37.195	325.42
b_{12}	-0.044	0.015	1	0.015	0.134*
Error	-	7093.02	4	0.114	-
Total	-	7960.71	7	-	-

* Insignificance at 5%

4.6 RESIDUAL ANALYSIS

Residuals from the 2^2 design were computed through a regression model. The relationship between the natural variables, the anodisation voltage and the anodisation time, and the coded variables is

$$X_1 = \frac{\text{Voltage} - (\text{Voltage}_{low} + \text{Voltage}_{high})/2}{(\text{Voltage}_{high} - \text{Voltage}_{low})/2}$$

$$= \frac{\text{Voltage} - (25+35)/2}{(35 - 25)/2}$$

$$= \frac{\text{Voltage} - 30}{5}$$

$$X_2 = \frac{\text{time} - (\text{time}_{low} + \text{time}_{high})/2}{(\text{time}_{high} - \text{time}_{low})/2}$$

$$= \frac{\text{time} - (40+50)/2}{(50 - 40)/2}$$

$$= \frac{\text{time} - 45}{5}$$

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Since the natural variable have only two levels, this coding produces ± 1 notation for the levels of the coded variables.

Thus if the voltage is at the high level (Voltage = 35 volts) then $X_1 = +1$ where as if the voltage is at low level (voltage = 25volts) , then $X_1 = -1$
Furthermore,
For high level of time (50 mins), $X_2 = +1$ and for low level of time (40 mins), $X_2 = -1$
The fitted regression model is

$\hat{U} = 32.074 + (10.189/2) X_1 + (2.156/2) X_2$

Where the intercept is the grand average of all 8 observations, and the regression coefficients b_1 and b_2 are one-half the corresponding factor effect estimates. The reason that the regression coefficient is one – half the effect estimate is that a regression coefficient measures the effect of a unit change in X on the mean of y and the effect estimate is based on a two- unit change (from -1 to +1)
This model is used to generate the predicted values of y at the four points of design.

Thus,
When the voltage is at the low level ($X_1 = -1$) and the time is at the low level ($X_2 = -1$), the predicted yield is

$\hat{U} = 32.074 + (10.189/2) (-1) + (2.156/2) (-1)$

$= 25.9015$

There are two observations at this treatment combination and the residuals are

$-e_1 = 19.55 - 25.9015 = -6.3515$

$-e_2 = 19.82 - 25.9015 = -6.0815$

For high level of the voltage and low level of time.

$\hat{U} = 32.074 + (10.189/2) (+1) + (2.156/2) (-1)$

$= 36.0905$

And

$e_3 = 40.0 - 36.0905 = 3.9095$

$e_4 = 40.3 - 36.0905 = 4.2095$

For the low level of the voltage and the high level of the time

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$\hat{U}= 32.074 + (10.189/2) (-1) + (2.156/2) (+1)$

$\quad = 28.0575$

And

$e_5 = 23.67 - 28.0575 = -4.3875$

$e_6 = 24.5 - 28.0575 = -3.5575$

Finally, for the high level of both factors

$\hat{U} = 32.074 + (10.189/2) (+1) \quad (2.156/2) (+1)$

$\quad = 38.2465$

$e_7 = 44.25 - 38.2465 = 6.0035$

$e_8 = 44.50 - 38.2465 = 6.2535$

4.7 RESPONSE SURFACE DESIGN

The regression equation obtained is

$\hat{U} = 32.074 + 10.189X_1 + 2.156X_2$

with X_1 ranging from 25-35 Volts and X_2: 40-50 mins.

The tables below were obtained from the regression equation to give yield (Thickness). varying Voltage and Time.

TABLE 4.9 YIELD VARYING VOLTAGE AND TIME

VOLTAGE (X_1)	TIME (X_2)	(THICKNESS)
25	40	373.04
26	40	383.23
27	40	393.42
28	40	403.61
29	40	413.80
30	40	423.98
31	40	434.17
32	40	444.36
33	40	454.55
34	40	464.74
35	40	474.93
25	41	375.20
26	41	385.38
27	41	395.57

28	41	405.76
29	41	415.95
30	41	426.14
31	41	436.33
32	41	446.52
33	41	456.71
34	41	466.90
35	41	477.09
25	42	377.35
26	42	387.54
27	42	397.73
28	42	407.92
29	42	418.11
30	42	428.30
31	42	438.49
32	42	448.67
33	42	458.86
34	42	469.05
35	42	479.24
25	43	379.51
26	43	389.70
27	43	399.89
28	43	410.07
29	43	420.26
30	43	430.45
31	43	440.64
32	43	450.83
33	43	461.02
34	43	471.21
35	43	481.40
25	44	381.66
26	44	391.85
27	44	402.04
28	44	412.23
29	44	422.42
30	44	432.61
31	44	442.80
32	44	452.99
33	44	463.18
34	44	473.36
35	44	483.55
25	45	383.82
26	45	394.01
27	45	404.20
28	45	414.39
29	45	424.58
30	45	434.76
31	45	444.95

32	45	455.14
33	45	465.33
34	45	475.52
35	45	485.71
25	46	385.98
26	46	396.16
27	46	406.35
28	46	416.54
29	46	426.73
30	46	436.92
31	46	447.11
32	46	457.30
33	46	467.49
34	46	477.68
35	46	487.87
25	47	388.13
26	47	398.32
27	47	408.51
28	47	418.70
29	47	428.89
30	47	439.08
31	47	449.27
32	47	459.45
33	47	469.64
34	47	479.83
35	47	490.02
25	48	390.29
26	48	400.48
27	48	410.67
28	48	420.85
29	48	431.04
30	48	441.23
31	48	451.42
32	48	461.61
33	48	471.80
34	48	481.99
35	48	492.18
25	49	392.44
26	49	402.63
27	49	412.82
28	49	423.01
29	49	433.20
30	49	443.34
31	49	453.58
32	49	463.77
33	49	473.96
34	49	484.14
35	49	494.33

25	50	394.60
26	50	404.79
27	50	414.98
28	50	425.17
29	50	435.36
30	50	445.54
31	50	455.73
32	50	465.92
33	50	476.11
34	50	486.30
35	50	496.49

Fig 4.1:Dependence of thickness on voltage and time of aluminum anodization

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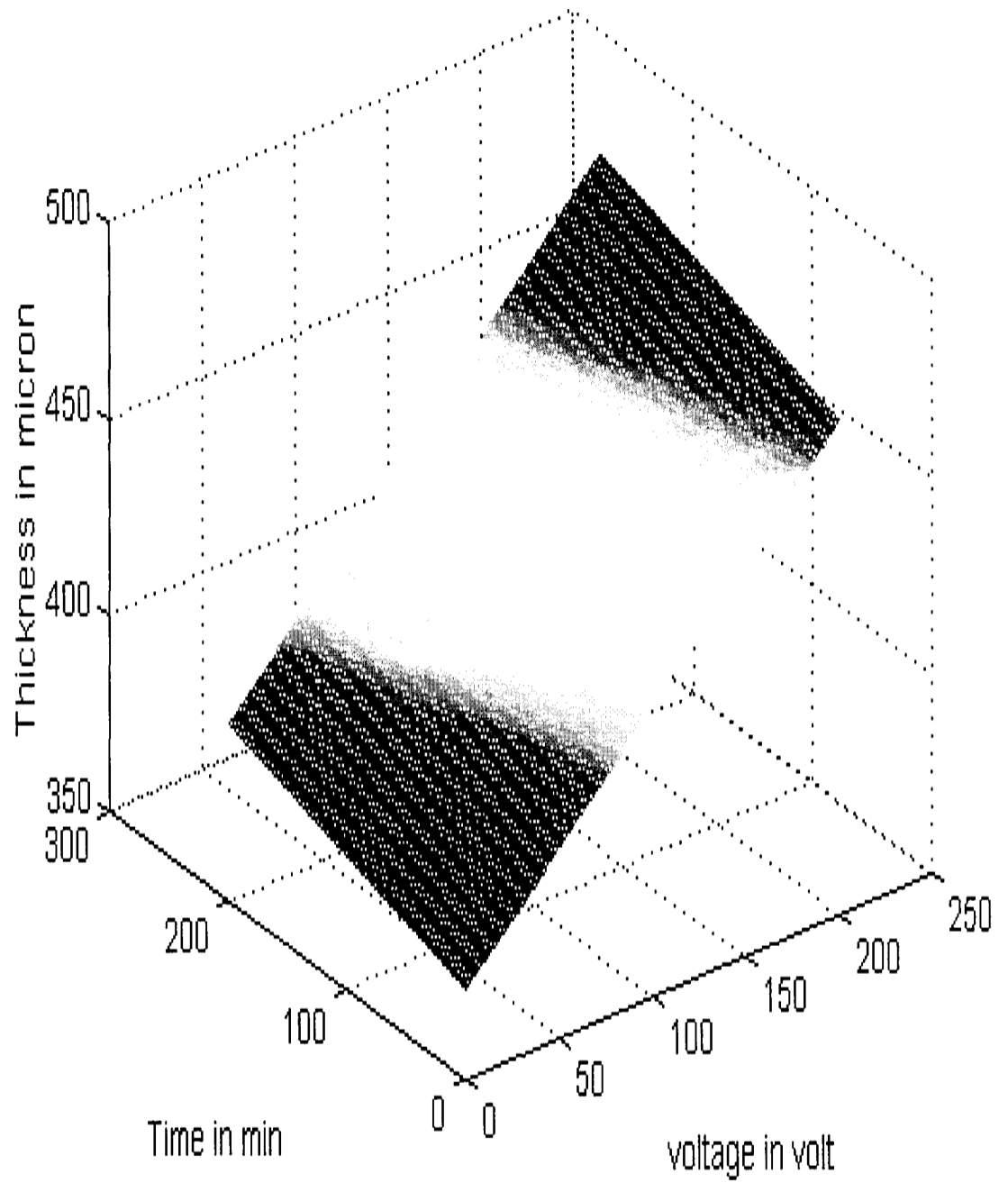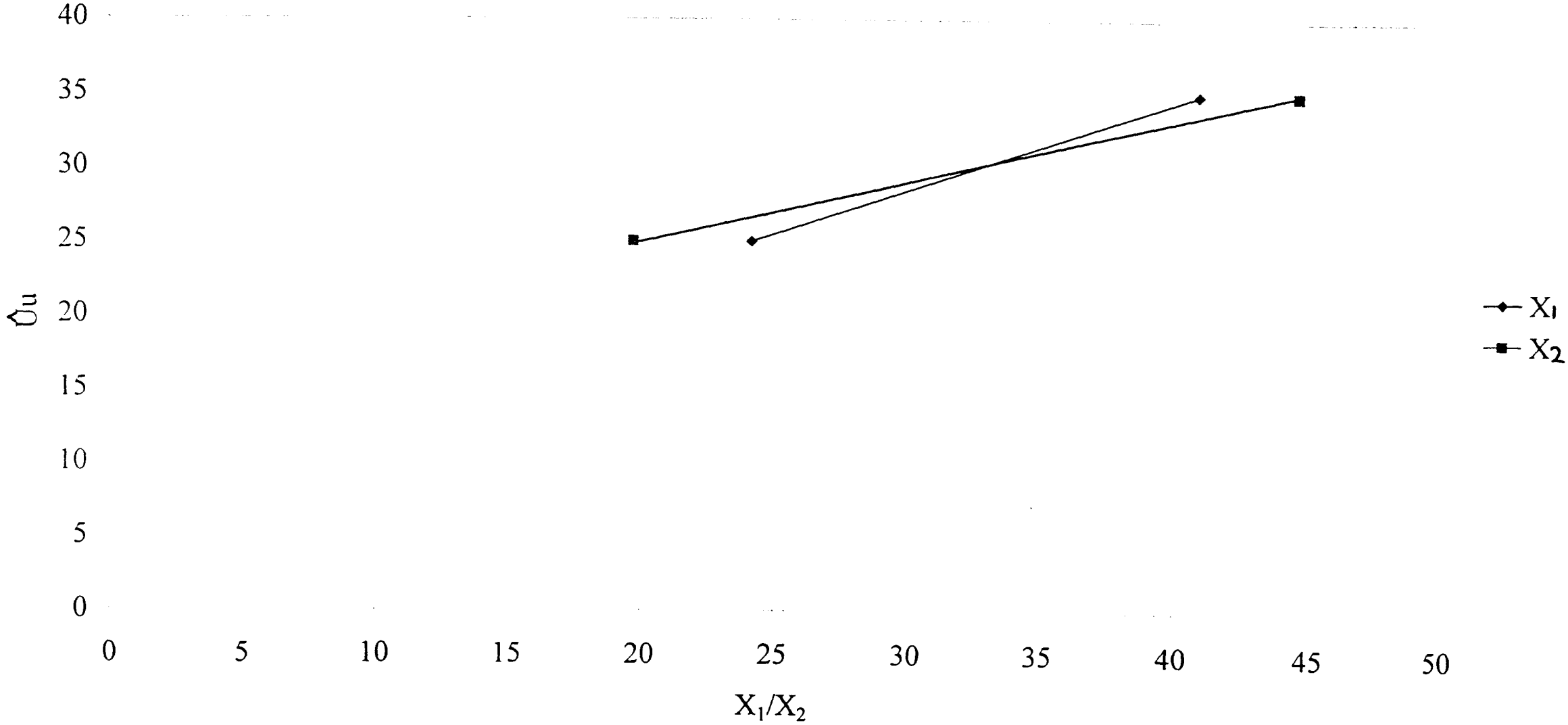
Fig. 4.2: Plot of significant main effect for anodisation of aluminium

5.0 DISCUSSION OF RESULTS

The corrosion resistance of anodized aluminium is particularly important in the building and shop front industries, and considerable work has been carried out to ensure that the coating will give satisfactory service. To achieve the best corrosion resistance the first requisite is adequate coating thickness. The coating thickness has a direct correlation to corrosion resistance with test indicating that the coating thickness is the most important single factor in corrosion resistance of anodic films.

Hence the dependency of the oxide layer formed by anodic oxidation on the conditions of the electrochemical process such as voltage and time of anodizing was investigated.

5.1   EFFECT OF VOLTAGE ON THE THICKNESS OF ALUMINIUM.

The thickness controlled at constant voltage depends on anodization time as shown in graph 1 where thickness is plotted against time and voltage. From table one of experimental data combined with the deduction from t-test and f-test, voltage was found to have a greater and positive effect on the thickness of oxide film on aluminum. Thus, increasing voltage from its low level (25Volts) to its high level (35Volts) increases the thickness of oxide film formed.

5.2   EFFECT OF TIME ON THE THICKNESS OF ALUMINIUM.

The effect of time is positive thus, this suggest that increasing the time of anodizing will increase yield (thickness of oxide film). Deduction from tables shows that as time was increased from its low level of 40minutes to its high level of 50minutes, thickness increased accordingly. Longer anodizing time produces a thicker film. Moreover, thickness is approximately a linear function of time.

5.3   THE COMBINED EFFECT OF VOLTAGE AND TIME ON THE THICKNESS OF ALUMINIUM

The interaction effect of voltage (A) and time (B) appears to be small relative to the main effect from the analysis of variance table, the interaction effect was shown to be

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insignificant at 5% degree of freedom. This was deduced from the statistical table of t-value and f-value. From the residual analysis the predicted values from the model equation shows that the highest thickness of anodized aluminium was observed when voltage and time were at the highest value respectively.

5.4 OPTIMIZATION OF THE ANODIZATION PARAMETERS.

Using the 2^2 factorial analysis, a linear response model was obtained as

$$\hat{U} = 32.074 + 10.189X_1 + 2.156X_2$$

The above model equation shows the relationship between voltage (X_1), time (X_2) and thickness ($\hat{U}$).

The response surface design shows graphically the relationship (Figure 4.1). The optimum thickness of anodized aluminium is difficult to find from the response surface (Figure 4.1).

This is because the surface is a plane.

CHAPTER SIX

6.0 CONCLUSION AND RECOMMENDATION

6.1 CONCLUSION

The result obtained is in line with findings of Douglas Montgomery. Thickness is approximately a linear function of time while the impressed voltage needed to sustain the action increases though not directly.

The developed model confirms that voltage and time determines the thickness of oxide film formed on aluminium though the optimum operating conditions could not be determined since the response surface is a plane. The model is valid only for values of X_1 and X_2 that fall within the interval of the values used in producing it.

6.2 RECOMMENDATION

1. All Engineers should receive formal training in experimental design as part of their undergraduate education because the successful integration of experimental design into engineering profession is a key factor in the future competitive of the industrial base.

2. This research work should be worked on in future in other to determine the optimum operating conditions and other parameters such as kind and temperature of electrolyte investigation.

NUMENCLATURE

A = Anodization Voltage

B = Time of anodisation

1 = All factors at low levels

a, b, = Factors at their high levels

X_1 = Coded value for A

X_2 = Coded value for B

Y_{u1} = Replicate 1

Y_{u2} = Replicate 2

$\hat{U}$ = Thickness

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CALCULATION OF THE MEAN AND DISPERSION

RUNS	REPLICATES		MEAN($\hat{U}_u$)	$|Y_{u1}-\hat{U}_u|$	$|Y_{u2}-\hat{U}_u|$
	Y_{u1}	Y_{u2}			
1	19.55	19.82	19.685	0.135	0.135
2	40.00	40.30	40.150	0.150	0.150
3	23.67	24.50	24.085	0.415	0.415
4	44.25	44.50	44.375	0.125	0.125

RUNS	$|Y_{u1}-\hat{U}_u|^2$	$|Y_{u2}-\hat{U}_u|^2$	S^2_u
1	0.018225	0.018225	0.03645
2	0.0225	0.0225	0.04500
3	0.172225	0.172225	0.34445
4	0.015625	0.015625	0.031250

$$\Sigma S^2_u = 0.0450 + 0.03645 + 0.34445 + 0.031250 = 0.45715$$

CALCULATION OF G-VALUE

$G_{cal} = 0.34445 / 0.45715 = 0.753$

CALCULATIUON OF MEAN SQUARED ERROR

From equation 3.5, the mean square error is calculated

Thus

$$S^2_y = \tfrac{1}{4}(0.45715) = 0.1142875$$

The experimental error is

$$S_y = \sqrt{S^2_y} = \sqrt{0.1142875} = 0.338064$$

TO CALCULATE EFFECT

The mean effects, b_o, was calculated from the effect of A & B at low levels

$b_o = \tfrac{1}{4}[(+19.686) + (+40.150) + (+24.085) + (+44.375)] = \tfrac{1}{4}(128.295)$

$= 32.07375$

APPENDIX B

The main effect, of A, b_1, which shows the effect of A at the low level of B and the effect of A at high level of B is

A = ¼ [(-19.686) + (+40.150) + (-24.085) + (+44.375)] = ¼ (40.755)

   = 10.18875

The main effect of B which is the effect of B at the low level of A and at high level of A is

B = ¼ [(-19.686) + (-40.150) + (+24.085) + (+44.375)] = ¼ (8.625) = 2.15625

The interaction effect AB is defined has the average difference between the effect of A at the high level of B and the effect of A at the low level of B

AB = ¼ [(+19.686) + (-40.150) + (-24.085) + (+44.375)] = ¼.(-0.175) = -0.04375

ESTIMATION OF STANDARD ERROR

Errors in each regression coefficient are the same, thus

$S_{bo} = S_{b1} = S_{b2} = 0.3380643/ \sqrt{4x2} = 0.119523779$

CALCULATION OF t- VALUES

t_0 = 32.07375/ 0.119523779 = 268.3461841

t_1 = 10.18875/ 0.119523779 = 85.24454368

t_2 = 2.15625/ 0.111523779 = 18.04034325

t_{12} = 0.04375/ 0.119523777 = 0.36603595

CALCULATION OF SUM OF SQUARES

SS_0 = 2/4 $(128.295)^2$ = 8229.8035

SS_A = 2/4 $(40.755)^2$ = 830.4850

SS_B = 2/4 $(8.625)^2$ = 37.1953

SS_{AB} = 2/4 $(-0.175)^2$ = 0.0153125

Total sum of squares, $SS_T =$

$[(19.55)^2 + (19.82)^2 + (40)^2 + (40.3)^2 + (23.67)^2 + (24.5)^2 + (44.35)^2 + (44.5)^2]$
$- [(19.55)^2 + (19.82)^2 + (40)^2 + (40.3)^2 + (23.67)^2 + (24.5)^2 + (44.35)^2 + (44.5)^2] / 8$

 $= 9097.95663 - 9097.963 / 8$

$= 7960.711763$

CALCULATION OF f- RATIOS

f-ratio of A $= 830.4850/0.1143 = 7265.84$

f-ratio of B $= 37.1953/ 0.1143 = 325.42$

f-ratio of AB $= 0.015313/ 0.1143 = 0.1339$