PRELIMINARY INVESTIGATION INTO

BLACKCOATING OF SHOE BUCKLES(BRASS)

USING LEAD ELECTROLYTE

BY

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DECLARATION

I Declare that this project report is an authentic report of my experimental work and that to the best of my knowledge has not been presented elsewhere for the award of any degree or diploma. All published work used has been duly acknowledge in the reference.

CERTIFICATION

This is to certify that this work "Preliminary Investigation into black coating of shoe Buckle (Brass) using Lead Electrolyte" was carried out by ADAH BENSON of the Department of Chemical Engineering, School of Engineering and Engineering Technology, Federal University of Technology.

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DEDICATION

This work is whole heartedly dedicated to God Almighty, the most merciful for his infinite care, strength and wisdom availed to me to go through this programme successfully.

This work is also dedicated to my Parent Mr. and Mrs P.O. Adah and my Kindreds for their boundless love and sacrifice

ACKNOWLEDGEMENT

First of all, I acknowledge God Almighty for his guidancce, grace, mercy, provision, protection against all devices of the enemy throughout the duration of my stay in this institution. It looked hectic and tedious at the inception but for his devined mercy, today I am through with the programme.

My sincere thanks to my project Supervisor, Mr. Duncan Aloko for his invaluable time spent to counsel and direct my project. Your role to me through my University Education is more like that of my parent. I pray God Almighty reward you abundantly.

I also wish to acknowledge all members of staff of the department of chemical Engineering for their unalloyed contributions to the success of my programme, most especially, the HOD, Dr. J.O.Odigure, Dr. Aberuagba, Mr Olutoye and others whose assistance has seen me through by programme.

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It will appear impartinent to conclude this degree programme without recognising distinguished, loving and dedicated friends that have really kept me company in Niger state, they are viz; Sgr.P.A. Bakka, Elijah Uzim, Chinyere Machie, Ismaila Musa, Alkali Bababuya, Fredrick Ayenigba, Joy Okotie, Salawu Moshood, Gabriel Adamu and lots more that time wouldn't permit me mention.

Finally, I duely acknowledge my brothers and Sisters; Adah Friday, Adah Kenneth, Adah Bright, Grace Adah.

I humbly pray that God Almighty kindly reward them abundantly amen.

For better optimisation of cost and output , it will be advisable to minimise the power consumption due to ohmic polarisation in the solution by increasing the throwing power of the bath, increase agitation to permit plating rate ,hence reducing the time of the plating. It is also advisable to operate at the maximum density in order to deposit on the cathode substrate at the shortest possible time .

ABSTRACT

The basic fundamentalities of the project was to ensure absolute protection on expensive, and scarce metal surfaces from corrosion, rust caused by CO_2, water droplet and oxygen. Others include achievement of brightened black colour deposition on the buckles and even thickness throughout the surface. To achieve the desired result the organic additives were added in relatively low concentration to enable the modification of the structure and morphology of the buckles.

The time for plating of the cathode was observed to increase with the voltage until an optimal voltage of 109 volts which correspond to a time of 45mins then the thickness of deposition was conspicious.

The experiment at the inception, requires the cleasing, pickling, degressing of the buckles to remove impurities of any sort; cleasing of this bath with the electrolyte solution. The d.c power source, a feedback control panel supplied a steady dc current of 0.5 Amperes. The current was constant so as to enable the decomposition rate of the anode material to be steady and uniform.

From mathematical calculation shown in the appendice, the best thickness of coating on the buckle was obtained in experiment 2 with a thickness of $1.5\mu m$ and the least thickened buckles are those for experiment 3&5 with value of $3.28\mu m$.

It can also be concluded that using the current market pricing of equipment specified in designing. The total cost of the major items of equipments is extimated to be N12,521.48k and the extimated total cost for the raw materialis calculated to be N18,660.00K.

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1.0   INTRODUCTION

Electroplating is the electro- deposition of an adherent metallic coating on an electrode in order to form a surface with the properties or dimension different from those of the basic metals. The term adherent excludes electroforming, which is usually considered a branch of electroplating technology and the term electrodeposition must be stretched to include electroless plating and immersion processes that do not employ electric current. Plating metal may be transferred to conductive surface (metals) or to non-conductive surface (plastic, wood, leather), after the latter have been rendered conductive by such processes as coating with graphic, conductive lacquer, electroless plate or vapourised coating

Actually, the practical and technology phases of electroplating matured much earlier than the science. Until about World War 1, electroplating remained essentially an art: solution composition were closely guarded family secrets, and most electroplaters made no pretense of knowing even the fundamentals of electricity or chemistry. Modern electroplating started in 1800 with A. Volta's discovery of the voltaic pile or battery, which made note worthy quantities of direct current electricity available. It was about the same time that the battery was employed to deposit lead, copper and silver. Most earlier uses of electroplating were predominantly decorative and the physical and chemical properties of the product were not so important as its appearance. The demands of world war 1 and especially II for finishes with close tolerances and rigidly specific properties converted electroplating from craft to a technology grounded in basic science and engineering. This development has been forwarded by advances in metallurgy and physical and electrochemistry, by improvements in sources of direct current and in measuring instruments and by the rise between the wars, of the chemical industry which made available reliable sources of pure chemicals.

Electroplating is a surface treatment. The substrate to be plated is made the

cathode is $Mn^+ + ne \rightarrow > M$ $\qquad$ (1.1)

At the anode, the reaction is the dissolution of the same metal ie

$M - ne \rightarrow M^{n+}$ $\qquad$ ....(1.2)

The electrolysis conditions are controlled in such a way that the current efficiency of reaction (1.1) and (1.2) are the same and hence the concentration of Mnt in the bath remains constant. In a few cases, the metal ion L.s to be added as a aslt and then an inert anode, usually lead dioxide, is employed; the anode reaction is then oxygen evolution.

For a successful electroplating process, the correct pretreatment of the cathode and careful selection of the anode material, plating bath, current density and other electrolysis conditions are essential. It has been observed to be common to electroplate:

I) single metals; the most important are Sn, Cv, Ni, Cr, tn, cd, pb, Ag, Au and pt.

ii)   Alloys, including Cu/Zn, C/Sn, pb/Sn, Sn/Ni, Ni/Co and Ni/Cr

iii)   Composites; ie metals containing solids such as PTFE or WC2.

The origin of the industry lay in the desire to enhance the value of metal articles by improving their appearance, but in modern times the importance of metal finishing for prely decorative reasons has decreased. The trend is now towards surface treatment which will impact corrosion resistance or particular physical or mechanical properties to the surface (e.g conductivity, heat or wear resistance, lubrication or solderability) and hence to make possible the use of cheaper subotrate metals to give them essential metallic surface properties. Other properties conferred on electroplating includes frictional characteristics, lucar resistance and hardness, specific electrical properties.

The objective of an electroplating process is to prepare a deposit which adhere well to the substrate and which has the required mechanical, chemical and physical properties. Moreover, it is of overrinding importance that the deposit properties reet

2.

many metals may, by modification of the bath and plating condition be deposited with different properties. The bath, current density, temperature, etc. will depend to some extent on the deposit properties required.

Owing to the importance of reproducibility in the deposit, it is important that the plating bath is stable for a long period of time. It is also necessary that the quality of deposit is maintained over a range of operating conditions since some varia;tion in concentrations and current density are periodically bound to occur particularly when different objects are to be plated. Tolerence of the bath to mishandling during operation on the factory floor is an additional advantage.

It is clearly desirable that the deposit has an even thickness over the whole of the surface to be electroplated. This requires the potential to be the same if all prints over the surface of the cathode and this is impossible to attain when the object to be plated has a complex shape-like shoe buckles. The evenness of the deposit can be improved by introducing auxiliary anodes (usually platinized titanium electrodes, where the reaction is oxygen evaluation) at various position in the electrolyte, the objective being to increase to cathode current densit at points where it would otherwise be very low ie at points from the cathode furthest from the normal anodes (e.g in holes or recesses in the object being plated).

The ability of the plating bath to give an even deposit is measured by its throwing power. The main process parameters which affect the throwing power are the composition of the plating bath (ie total electrolyte concentration, complexing agents, PH, additives), temperature and current density. From the view point of energy efficiency it is attractive for the electrodeposition process to have a high current efficiency and the plating cell to be designed to have a low anode-cathode voltage. The energy used in the electrolysis itself is commonly low compared with that required to heat the bath and to prepare the surface prior toplating. The electroplating process of many metals is carried out with a very high current efficiency with the exception of chronium deposition.

heavy-metal and transition - metal ions. Thus all large electroplating facilities will have a plant for the removal of metal ions and other toxic species before effluent water is discharged.

1.2.0 SCOPE OF WORK

1.   Use local methods and technology to achieve black coat.

2.   Use electronical process to achieve deposition which adhere well to the substrate.

3.   To give the coated material durability and present corrosion on the metal caused by CO_2, H_2O and oxygen.

4.   To achieve a brightened black colour deposition on the buckle and even thickness throughout the surface.

1.3.0 LIMITATIONS

This work is limited to ensuring the technical importance associated with the process of electroplating. The basic features to be observed include; achieving brightened black colour deposition on the buckle, an even thickness throughout the surface of the buckle and produce level deposit on a microscopic scale.

In course of the laboratory work, an acceptable technical objective was obtained at an optimal voltage on 109 volts and a constant current of 0.5 Ampers, the current density for the anode and cathode was maintained at 300 Aldm2 and the temperature of 40^0c.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 BATH PREPARATION

The first step in preparing a new bath consists of thorough cleaning of the empty platire, tank. All solid particles and dirts are first removed by sweeping and wiping. If the tank contains grease, it should be wiped out with rag containing a suitable solvent. Rubber or plastic tanks require no further cleaning, where as wooden tanks may require painting of lining with pitch.

For electroplating, cleaning, rinsing, degreasing, picking are most important which are described hereunder:

2.1.1 CLEANING

The cleaning operation is carried out in some type of alkaline cleaner. The type selected depends on the material to be cleaned and on whether the cleaning is to be electrolytic or by soaking. Cleaning may contain sodium phosphate, sodium hydroxide, sodium silicate, sodium carbonate, sodium borate or any combination of these salts. Most cleaners consist of phosphate or silicate with some hydroxide or carbonate added. Electrolytic cleaners may be operated with the work as the anode or the cathode. Anodic cleaners are often preferred because metal impurities cannot deposit on the work.

2.1.2 DEGREASING

It is advantageous to degrease prior to electrolytic cleaning. Degreasing may be done by dipping the work in a bold solvent or it may be done by exposing the work to hot vapour. The vapour will condense on the metal and only clean solvent will come in contact .with the work.

2.1.3 PICKLING

The plating process consist of some type of etching which is required to remove inorganic surface films and expose the metal to the action of the plating bath. If this step is not carred out properly, peeling iblistering or poor adhesion of the deposit will result. Ectching is usually done with mineral acids, sulphuric being preferred and hydrochloric is also used. Pickling may also be done electrolytically, the advantage being that the solution of metal can be treated by the current and the amount of metal removed can be more closely controlled.

2.1.4 RINSING

Rinsing is generally done in running water but it may also be done effectively by spray.

2.2.0 SUBSTRATE PREPARATION

For a useful electroplate to be deposited on the surface, it must be conditioned to receive it, that as the surface must be prepared as to ensutre the desired bond. A practical definition, then of a satisfactorily clean surface would be a surface containing no foreign material that interferes with the formation of an adherent deposit. Hence, this connotes the removal of gross dirt and soil, heavy oxide or tarnish films.

A typical cleaning cycle includes the following:

I)   Pickling to remove gross scale.

ii)   Any mechanical preparation such as polishing or buffling

iii)   Cleaning to remove oils, greases, shop dirt and polishing and buffling comuponds

iv)   Rinsing

v)   Acid dripping to remove oxide films and

vi)   Rinsing

used to bond buffing and polishing compounds. The problem associated with using an organic cleaner is .that removal of oils and greases from a highly contaminated surface may merely leave the non oily dirt dry and more firmly attached than ever. Common solvent for this purpose are perctlorocthylene, trichloro ethylene, trichloro trifluoroethane,methylene chloride and methyl chloroform.

Alkaline deaning generally follows the organic cleaning and its function is to dislodge surface soil, principally by dispersing that it does not settle back on the work and to some slight extent by saponifying the saponifiable oils and greases. Alkaline cleaning may be accomplished by one of the three techniques;

I)   Soak

ii)   Cathodic or direct

iii) Anodic or reverse

Alkaline cleaners are invariably applied at elevated temperatures. Soak cleaning the part involves immersion on the cleaner and with mild agitation. In direct cleaning, when the work is the cathode twice as much gas, in this case hydrogen is evolved as when the work is the anode (reverse cleaning) in which the gas revolved is oxygen. In cathodic cleaning the impurities in the cleaners may be deposited on the surface or hydrogen may embrittle the basic metal. Anodic cleaning, on the other hand may form oxides on the surface of susceptible materials.

The water-break test is mostly used universally as test of adequate cleaning. Water break is the gathering of a.film of water into depplets or streaks and its occurrence is a sign that the surface is not clean.

Acid dipping generally follows cleaning and serves two purposes:

I) ·   Removes slight tarnish or oxide films formed in the cleaning step.

ii)   Neutralises the alkaline film.

Acid dipping is usually a 10 - 30 vd% solution of Hcl or H_2SO_4, preferably

2.3.0 THE ELECTROPLAITING PROCESS

The operation of electroplating, including the cleaning, rinsing, plating and post plating treatments can be carried out manually or with almost any desired degree of automation. Parts may be hung in the plating tank or wires or on racks; when many small parts are to be ploted, they may be contained in wire baskets or more commonly in barrels that rotates in the plating tank. Movement from one operation to the other may be by hand or by machine.

The necessary d.c power is derived from motor generator or increasingly by a feed back control system. Power is conveyed to the plating tanks by bus bars; the anodes are hung in the tank from the positive bus bars and the work to be plated from the negative cathode bar, usually down in the centre. Tank voltage is read from the voltmeter, and current from an ammeter. A thid instrument, an ampere-hand meter is often helpful a means of regulating the thickness of deposit and for general control of the operations.

Some agitation of plating solutions or of the work is usually helpful. The oldest and the simplest method consists of an operator merely switching the work around at intervals, but automatic cathode rod agitation is preferable. In some automatic plating machines the work is moved through the solution while it is plated; provided clean air is used and the solution is not sensitive to oxygen or carbon dioxide. Air agitation is convenient and efficient. Agitation may be provided by stirring or pumping the solution, the latter is often necessary when heat exchangers are used for temperature control or where continuous filteration is required. In barrel plating, agitation is supplied by the movement of the barrel.

Temperature control is almost desirable in plating operations because the characteristics of plating solutions, of the deposit or of both usually depends to a large extent on the tempera;ture of operations. Heating or cooling coils in the tank itself may be used, or the solution may be circulated through a heat exchanger. Electric immersion heater may be used too.

other precautions should be taken against stray currents, which may be very troublesome and extremely difficult to trace. In addition to the basic equipment; flters for either continuous or intermittent purification of solutions; drying facilities, which may range from a simple jet of compressed air to large ovens, racks of design appropriate. To the part being plated, and a racking station where the work can be conveniently hung on these racketr and unracked after plating; one or more stripping tanks for stripping faulty deposits so that part can be reworked and for stripping the plating rack themselves; reclaim tanks if the drag-out is valuable and worth reclaiming; portable pump for transferring solutions; and at least one empty tank so that the plating tank can be emptied and worked on; apparatus for waste treatment, metal recovery, or both.

Each electroplated metals will require somewhat different deposit thickness and characteristics and hence a different bath formulation and electrolysis condition. For several metals there is a choice of acidic, non complexing bath which is used for rapid plating of simple shapes e.g flat surfaces and wires and a complexing medium which gives a lower plating rate but the additional throwing power assentral for introcate surfaces. Most plating processes operates at a current density between 10 and 70 Macm2, a value which is low compared with that for the preparative electrolytic processes. In general, the thickness of the deposit required will range between 0.01 and 100mm depending on the application. The electroplating procedure will only last from a few seconds to thirty minutes. The current density for silver deposition is particularly low while those of wickel in sulphumate media and tin in the chloride/flouride bath are markedly higher. Most of the current is hydrogen evolution. For most metals current efficiencies are good and that the use of additives and temperature slightly above ambient is normal.

The plating of some alloys is also possible although the limitations are greater and the control of the conditions is more critical. The alloy deposited clearly depends on the electrolyte, bath temperature, current density and the ratio

towards the low end of the metal plating range and many of the alloys do not dissolve .anodically so that it is necessary to use anodes of both metals.

When very small objects such as screws, nuts and bolts are to be plated, it .is usually carried out by barrel plating. They are loaded by the weight into a perforated barrel maid either from metal, when it also acts as the anode connection or from plastic, when cathode contacts are placed inside the barrel. The barrel is rotated slowly (5 - 20 revolutions per minute) in the various solutions and in the plating step an even deposit is obtained to lure random orientation with respect to the anode.

The electroplating step is normally carried out in a simple rectangular tank of volume between 10 and 20 litres. They should be lagged and have provision for heating the electrolyte. It is also frequently advantageous to introduce convertion into the plating tank and where oxygen does not interfere with the metal deposition, bubbling air through the electrode is the cheapest procedure. The sludge, placed symmetrically about the cellove jig; the position of the jigs, man anode and auxiliary anodes may be based on experience or detailed computer optimisation depending on the cost of the product and the scale of the operation.

In large facilities, the tanks will have a loop for continuous filtering of the electrolyte and the bath temperature, PH and current will be monitored. The concentration of metal ions will be determined by external analysis on a regular basis. The most modern plants will also have an efficient control plant to deal with liquids resulting from washing and spillages from the tanks.

The heavy metals are commonly precipatated with caustic soda and the solids then allowed to settle. The control units for the electroplating baths operate at a current between 100 and 10000 A and a voltage of -8 to -12V.

2.4   PLATING BATH CONSTITUENTS

The plating bath is normally a complex mixture of salts of the metal being plated, electrolyte, and various additions to ensure that the eletroplate has the desired properties and quality. The constituted compoents include:

2.4.1 THE METAL ION

The metal to be plated is present in solution either as the simple hydrated ion or as a complex but always in high concentration, typically 1.0 - 3.0m. This high concentration is essential because, while it is necessary to use a reasonable current density, the quality of the plate suffers badly if the deposition occurs under conditons where the electrode reaction is even partially mass - transfer - controlled.. A non complexing medium is frequently used for rapid plating on objects of simple shape while complexing media are employed when a high throwing power is of importance.

2.4.2 ELECTROLYTES

Electrolytes are also added to in high concentration to give the bath maximum conductivity. They also have the role of controlling the PH and buffering the solution particularly if hydrogen or oxygen is evolved at the cathode or anode respectively, since this reaction will tend to change the PH. An increase in PH may lead to deposition of metal hydroxides or a decrease of H2 evolution.

2.4.3 COMPLEXING AGENTS

Complexing agents are used to make the deposition potential more negative when it is necessary to prevent a spontaneous chemical reaction between the cathode and the plating ion. e.g plating copper onto iron or steel.

$$Cu2+ +Fe --> cut Fe 2+ \qquad .... 2.0$$

This chemical reaction will lead to a very poor copper deposit so a complexing

agent is added to rack the potential of the Cu2+/Eu couple negative to that for the Fe/Pe 2+ couple; then equation 2.0 is then no longer thermodynamically favourable. Complexing agents are. Used to improve the throwing power of the bath. The most common complexing agents I electroplating are cymide, hydroxideand more recently, sulphamate ion.

Complexing agents also have a role at dissolving anodes; they can prevent passivation and therefore, loss of current efficiency in the corrosion reaction and it is for this reason that a low concentration of chloride ion is a common constituent of many baths.

2.4.4 ORGANIC ADDITIVES

A wide range of organic molecules are added in relatively low concentration to the electroplating bath to modify the structure/morphology and properties of the cathode deposit. Their development has been almost totalling empirical and details of their mode of operation are seldom known. Generally additives are usually capable of absorption on the cathode surface and in some cases organic matter s included into the deposit, especially when the plated metal has a igh surface energy (high melting point). Many additives also increase the deposition over potential and this may be due to the need for electron transfer to occur through the absorbed layer or due to complex formation at the electrode surface.

There is clear evidence that when several additives are present in the electrolyte, their effect is synorgetic and significantly improves the technical properties as classified below:

2.4.4.1 BRIGHTENERS

I obtain a bright deposit, the microscopic roughness of the deposit must be lead compared with the wave length of the incident light so that it is reflected

rather than scattered. Brightness are commonly used in relatively high concentration (gL-1) and may result in substantial organic matter in the deposit. They usually cause the formation of an even, fine-grained deposit and hence may set by modification of the nucleation process.

Two types of brighteners are recognised, the first are aromatic sulphones or sulphonates and they lead to bright deposits without lustre and also reduce stress. The second are molecules containing $- C \equiv N$, $\sim N - C \equiv S$ or $C = O$ entities eg thiorea and coumarin, which produce deposits with a high lustre but also raise the stress and brittleness in the metal.

2.4.4.2 LEVELLERS

These produce a level deposit on a mere microscopic scale and get by absorption at points which otherwise there would be rapid deposition of metal. Thus absorption of additives occurs preferentially at dislocations because of a higher free energy of absorption and at peaks because the rate of their diffusion to such points is enhanced to absorbed additive will reduce the rate of electron transfer.

2.4.4.3 STRUCTURE MODIFIERS

These additives change the structure of the deposit and may be even the preferred orientation or the type of lattice. Some are used to optimise particular deposit properties and others to adjust the stress in the deposit (stress is due to lattice masfit). The latter are called stress relievers.

2.4.4.4 WETTING AGENTS

These are added to accelerate the release of hydrogen gas bubbles from the surface. In their absence, the hydrogen which is often evolved in a parallel reaction to metal can become included in the deposit cayusing, for example hydrogen embrittlement.

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combined with the genrally good resistance of nickel to many corrosive chemicals, makes it useful in a host of industrial applications. Worned or mis-machined parts can be built up to proper dmensions by nickel or chromium plating, such expensive equipment is thus salvaged at a small fraction of the cost of replacing it.

Bearing properties can often be greatly improved by electroplating; porous chromium serves this purpose. Aluminium alloy pistons for internal combustion engines are tin plated to prevent scoping of the cylinder walls by the abrasive aluminium oxide during the running-in period.

2.9.0 ELECTROFORMING

Metals used for electroforming constitute few of these before atomic number 26 (iron). Thus aluminium is frequently deposited in this way from organic or non-aqueous solutions and also fused salt baths. Some of the refractory mtals (molybdonum, tungsten, tantalum and niobium) have also been applied n this way by workers using fused (melted) salt baths. In electroforming, the electrodeposited metal is usually much thicker than for other applications up to 0.6cm or more.

Success in electroforming depends primarily on two factors: extreme care in the plating operation, and skill and foresight in the design of mandrels to ensure their successful removal from the form. In this manufacturing technique metal is deposited upon a form or mandrel which is later separated from the deposit mandrels for electroforming are of two types: temporary, those used to produce one electroform and then removed by melting or dissolving away the electroform; and permanent, those that are removed from the electroform intact. Choice between them depends on shape of the electroforms and the number of pieces to be produced. Temporary mandrels includes aluminium or zinc that can be dissolved away chemically while permanent mandrels are usually of stainless steel, chromium plated steel, nickel, alloys, or aluminium.

2.9.1 IMMERSION PLATING

Involves the replacement of a metal ion in solution by an atom of the substrate. For example, tin can be plated on copper in this manner. Immersion tin on coper is .used in electronics and printed circuit boards. Immersion tin is also used for plating the inside of copper pipes.

2.9.2 ELECTROLESS PLATING

Electroless plating is a process of plating by chemical reduction. For example, nickel can be plated by the addition of a reducing agent, such as sodium hypophosphite, a solution to nickel salts. The nickel ion is reduced to the metal, which under proper conditions is deposited as a coherent and adhesive plate rather than simply as a powder throughout the solution. The reaction takes place on specific catalytic surfaces and the process is more expensive than electroplating. Electroless plating can be used where electroplating is impossible, example plating the insides of pipes. An increasingly important use of this process is for plating nonconductors, particularly plastics. The plastic surface first treated with sensitizers and activators.

2.9.3 ELECTROMIGRATION

Electromigration or electrotransport refers to the net motion of atoms owing to the passage of an electric current through a metallic conductor in either the solid or liquid state. Simple metals may be considered to be constituted of atoms stripped of their valence electrons, existing as ions surrounding by a sea of electrons. In the presence of an electric field, the ions cores are subjected to a force directly resulting from the field, as in an electrolytic solution. In electromigration there is, in addition to this direct field force, a force resulting from the friction between the numerous and rapidly moving electrons and the ion cores. This force is known as the electron wind force, is in the opposite direction

to the field force in electron conductors and for metals in electrolytic solution, the current results entirely from motion of ions, in usual electromigration phenomena the fraction of the current contributed by ion motion is quite negligible in comparison to that fraction of the current caused by the motion of the charge carriers, either electrons or holes.
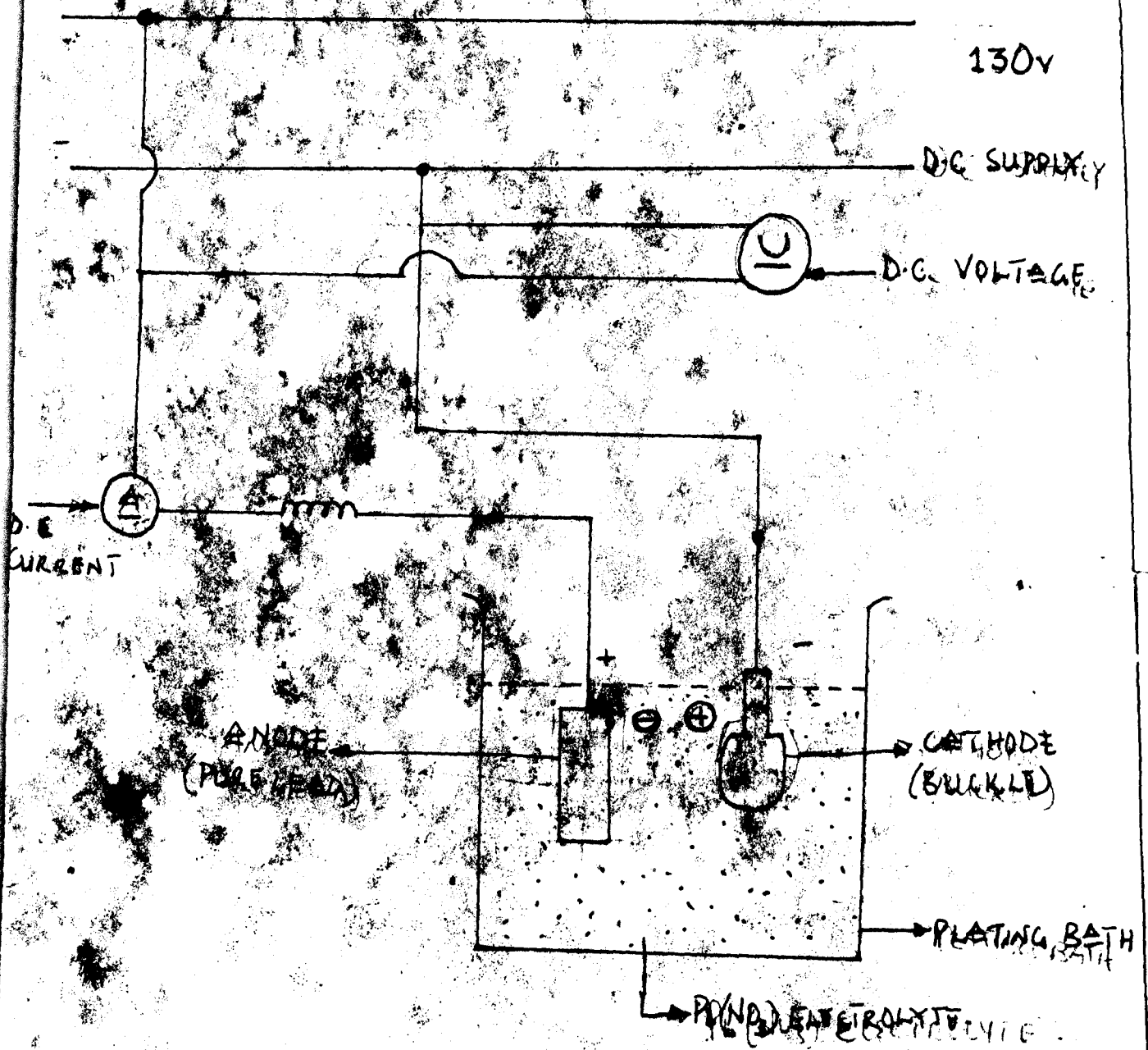
130v

D.C. SUPPLY

D.C. VOLTAGE

D.C CURRENT

ANODE
(PURE LEAD)

CATHODE
(BUCKLE)

PLATING BATH

PbN electrolytic

FIGURE 11 DIAGAM   SHOWING CIRCIUT CONNECTION AND
FLOW OF ELECTRONS

xx) Distilled water   7 litres

xxi) Empty bottles   14000cm^3

3.2 PREPARATION OF SOLUTION TO SPECIFICATION

3.2.1 PREPARATION OF PB (NO3)2 SOLUTION

The required 97grams of the substance was weight with an electric balance and pured into a beaker. Distilled water of volume 1000cm3 was measured with a cylinder and pured into the beaker containing the $Pb(NO_3)_2$ salt.

The mixture was thoroughly mixed with a stirrer until the salt dissolved completely. $Pb(NO_3)_2$ is the major electrolyte for the electroplating process.

3.2.2 PREPARATION OF K2CO3 SOLUTION

35 grams of K_2CO_3 was measured with an electric balance. 1000cm3 of distilled water measured with the cylinder was poured into a beaker contining the K_2CO_3 substance. K_2CO_3 is a unique additive in this experiment that modify the structure, morphology and properties of the cathode deposit.

3.2.3 PREPARATION OF KOH SOLUTION

250grams of KOH pellete was measured with an electric balance. Because of it hydroscopic nature, the measure 1000mc3 distilled water was introduced immediately. KOH is one of the additives used for electroplating.

3.2.4 PREPARATION OF PHENOL SOLUTION

2grams of phonel was diluted with 1000cm3 of distilled water in a beaker. Pure phenol is a colourless solid melting at 42^0c, moderately soluble in water and weakly acidic phenol reacts readily with alkali hydroxide to form salt.

$C_6H_5-OH + KOH \longrightarrow C_6 H_5 DK + H_2O$
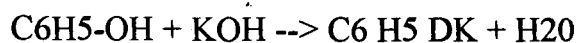
Phenol is used for surface active agents, other uses includes, lube oil additives

2.5

gasoline additives, drugs etc.

3.2.5 PREPARATION OF H2504 SOLUTION

2.72ml of concentrated H2504 was diluted in 1000cm3 distilled water. It is a colourless and has a densith of 1834 at 25^0c. The acid decomposes the salt of the main electrolyte, thereby allowing decomposition of the anode (P6).

3.2.6 PREPARATION OF GLYCERINE SOLUTION

Glycerine is the simplest trihydric alcohol with formular CH2OHCHOHCH2OH. It reacts to give the important class of products known as alkyl resin which is widely used as coating and in paints. Pure glycerine is colourless, odourless, viscous with sweat taste. It is completely soluble in water and alcohol. It boils at 2500c at atmospheric pressure and melts at 179^0c. It specific gravity is 1.26 at 25^0c and molecular weight of 92.09. 65ml of the liquid glycerine was diluted in 1000cm3 distilled water.

3.3 EXPERIMENTAL PROCEDURE

The main electrolyte and the additives were mixed accordingly to specification with distilled water. The feed back control system was the source of dc power aimed at supplying a constant current of 0.5 amperes at an optimal voltage of 130volts.

For cathode material (ie buckle) was thoroughly cleaned to ensure absence of gross dirt, leaving wide or tarrish films, by pickling, rinsing, and dipping and by mechanical preparation such as polishing. Furthermore, the plating bath the beaker of 250ml was thoroughly washed and rinsed with the main electrolyte. The anode material pure lead was harmmered until flattened. This is to ensure easy decomposition of the anode material in the electrolyte solution.

100ml solution of the main electrolyte Pb (NO3) was measured into the plating bath and a subsequent addition of 20ml each of the other additives in the order of

K2C03, KOH, phenol, glycerine and H2504 was done. The mixture was mixed well by stirring it. With the aid of the connecting wire, the anode material was connected to the positive end of the feedback dc power source through the variable rheostat and the ammeter. The variable rheostat was varied to maintain a steady current of 0.5 amperes, while the ammeter justify the required amount of current. The material to be plated, the shoe buckle is made the cathode and connected to the negative end of the feedback control panel. Meanwhile, each buckle was weight before commencing the experiment.

The Avometer which measure the voltage across the system is connected parallel to the process and it measured the varying voltage within the minimum range of minutes using a stop watch and at an interval of 5 minutes. On putting on the power source, it was observed that after 10 minutes, decomposition of the anode began and deposition on the cathode material became visible through te electrolyte different voltage. Values were obtain at different time intervals. Five experiments were carried out and it was observed that the optimal voltage value for proper coating is 109 volts at 95°c temperature.

After the completion of the experiment the power source was put off immediately on ensuring that the other apparatus indicator points at zero, the cathode material was removed from the electrolyte solution and collected in a beaker containing water which was later decanted. The buckle was dried in an electric ovum at a temperature 120°c for 55 minutes before reweighing was repeated.

Th experiment continued this way, although varying the quantities of the additives, main electrolyte Pb(NQ3)2.

2.5.1 CURRENT DENSITY RANGE

Current density is the average current in amperes divided by the area through which that current passes; the area is usually nominal area. Current densities at the anude and cathode are bot important; they may differ considerably although not by so large a factor as to make the necessary bery great difference between the anode are and the cathode area. Too high a current density at the anode may render it passive and too low current density may cause the metal to dissolve in an unwanted valence state. All thing being normal, the higher the current density obtainable, the faster the plating rate and thus, in general, the more economical the operation.

2.5.2 CURRENT EFFICIENCY

Paraday's laws predict the amount of metal that is deposited at the cathode or dissolved at the anode, but these amount are not always realised, the deficiency is due to evolution of hydrogen at the cathode of oxygen at the anode, it is often important to analyse the efficiencies at cathode and anode so that the bath remains in balance.

2.5.3 TEMPERATURE

Control of temperature is important in almost all plating processes since each solution is characterised by a range of temperature within which it gives the best results. Temperature affects all the variables of the solution; conductivity, current efficiency, nature of the deposit and stability.

2.5.4 ANODES

Anodes in a plating bath perform two functions, they act as the positve electrode, introducing current into thwe solution, and in most cases, they replenish the metal deposited at the cathode, thus maintaining the balance of the bath. The

performance of the anode is of considerable impotance in determining the success of an electroplating process. Where possible the anode is made from the same metal as that being plated at the cathode and the conditions are selected so that the concentration of the metal ion in solution remains constant. This requires the anode and cathode current efficiencies to be matohed (usually with values approaching 100%) over the whole service life of the anode. In particular the electrolytes in the plating bath must be chosen so that the anode does not passivate. In the active region, the electrode reaction is the desired metal dissolution but on increasing the current density or the over-covered with an oxde film which passivates the anode, that is, causes the current for the corrosion reactopm tp drp[tp a low level.

In a constant - current electrolysis, the potential of the anode will shift into the transpassive region where oxygen evolution is a major component of the current. Hence, n a plating process, it is essential to maintain the anode potential in the active region.

2.6 THE MECHANISM OF THE ELECTRODEPOSITION OF METALS

The deposition process has two distinct phases. At the start of the plating process, the deposition of the metal will occur on an electrode surface of the different material, that of the object to the plated. The first step will be the formation of nuclei of the new phase and their growth into crystals with the characteristic lattice of the electroplating metal. Once the electrode is fully covered, one is dealing with a M/M^m electrode and the thickening of the layer into a microscopic deposit. In terms of time or charge, the plating process consists almost entirely of the latter process, bu the initial stages are also. Important since they determine the structure of the primary layer (such as the shape and number of growing crystallites) and thereby influence the structure of the final electroplate and its properties. The density of nucleation is dependent on the plating bath and

is extremely sensitive to the electrode potential and alsothe current density.

The growth of the nuclei formed occutrs quire rapidly at comparatively low overpotentials, and in a constant current situation the potential will decrease substantially once nucleation has occurred. The growth of the crystals occurs by incorporation of the individual metal atoms into the crystal lattice. The newly incorporated metal atoms is only likely to be stable when it enters the lattice at a site where it interacts with several other atoms already in the lattice).

The overll phase growth is a sequence of at least three steps:

I. Mass transport (this may be diffusion, convertion, or migration) of the metal-bearing species to the electrode surface.

ii. Electron transfer to form an adatom

iii. Diffusion of the adatom across the surface into a position in the lattice.

The structure of the growing layer will largely be determined by the relative rates of processes, the current density employed. At low current density, the surface diffusion is fast compared with electron transfer. At high current densities, surface dimension is no longer fast compared with electron diffusion transfer and further nuclei must form. The rate metal deposition reaction that effectively occurs at an electrode of the electrodeposited metal is

$$L = nFK^{-D} (Cm^{nt} + exp (-\alpha nF/RT \wedge))$$

2.7.0 PRINCIPLES OF ELECTROPLATING

Electricity is a cheap reducing agent in electroplating, so for economy it should be used.

2.7.1 PRESENCE OF ION AND ELECTRICAL CONDUCTANCE

When an electric field is applied to a metal, according to the direction of applied field, electrons starts moving from negatively charged and of metal to its positive end, this way current flows in metal. Thus, a substance conducts

electricity when an electron or some other electrically charged particles like ion have to move through it.

In all electrically conducting solution, the solute molecules give rise to two types of electrically charged particles or ions. The negatively charged type of ions are called anions and the positively charged ones are callec cations.

2.7.2 CATHODE AND ANODE

The metal plates dipped in a solution through which the electrons are librated or absorbed, are called electrodes. The electrode which liberates electrons from the solution after acquiring them from negatively charged ions in the solution is positively charged, and called the anode, but the electrodes which gives electrons to the solution (negative charged) is called the cathode.

2.7.3 ELECTRICAL CONDUCTANCE OF A SOLUTION AND CONCENTRATION OF IONS IN IT

When an electric current is passed through a metallic conductor, according to the direction of the applied electrons flow as a steam in a single direction. Thus, flow of current in an electrolyte solution have two streams of cations and anions flowing in mutually opposite direction, but the total current strength is the sum of the current strengths due to the streams of cations and anions.

2.7.4 ELECTROLYTES

The electrolyte is the medium that carries the current by means of ions. The abilioty of a solvent especially water to ionize substances dissolve in ti, such that to split them into two components which carries negatively and postively charged that enables electroplating possible.

2.7.5 ELECTROLYSIS

A solution which is a good conductor of electricity has cations and anions in it. These ions are normally produced from solute and solvent. When an electric current is flown in solution by dipping electrodes in positively charged ions (cation) move to the negatively charged electrode and negatively charged (anions) moves to the positively charged electrode at the electrode. Thus, in an electrochemical reactio, the electrochemical changed involving combination with release of electrons takes place and ions are normally converted to the electrically neutral product which are totally different from solution. This phenomena is called electrolysis.

In other words, electrolysis is a process in which the anions and cations present in dissolved state of a substance are applying an electric field, these are transferred to their respective electrode (ie cathode and anode), and these loses there charges and are converted to new substances.

2.7.5.1 FUNDAMENTAL LAWS OF ELECTROLYSIS

Products of electrolysis only appear at the electrode surfaces and no where elese in the solution, because the electrolysis is the result of electron acceptance and electron shadding phenomena, and thus can occur only at electrode surfaces.

2.7.5.2 FARADAY'S FIRST LAW OF ELECTROLYSIS

In any process of electrolysis, the deposits formed on an electrode is directly proportional to te fusalit of electricity flown through the electrolyte. The quantity of electricity passed through the electrolyte during electrolysis is proportional to the current strength and for the time for which current is passed.

If the quantity electrity (Q) is measured in columns and strength of the current (I) measured is ain ampere and time (t) is measured in seconds, then Q

$$Q = I \times t$$

weight of deposit on electrode (W)

$$W = ZIT$$

where Z is the constant of proportionality and depends upon the characteristic of the ionic species discharged at the electrode. The constant is called electrochemical equivalent of the ionic species and it unit is gram per coulomb.

2.7.5.2   FARADAY'S SECOND LAW OF ELETROLYSIS

States that, when the same quantity of electricity is passed through a number of electrolytes, the weight of corresponding deposits at differet electrodes are exactly proportion to their chemical equivalents respectively.

Chemical equivalent or

$$\text{equivalents weight} = \frac{\text{atomic weight}}{\text{Valency}}$$

By second law, when a current is passed through two electrolyte, if the deposit in two different electrolytes, electrode is W1 and W2 gm their equivalent weight is M1 and M2 then,

$$\frac{W1}{W2} = \frac{M1}{M2}$$

2.8   ENGINEERING APPLICATION AND TECHNICAL IMPORTANCE

Application for plating are classified according to the principal function of plate:

I.   Appearance and adhesion

ii)   protection against corrosion

iii)   Special surface properties

iv)   Engineering or mechanical properties

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2.8.1 APPEARANCE AND ADHESION

The list of articles plated for appearance is almost endless, automative, aicraft parts, refrigerator, hardwares electrical appliance, plumbing fixtures, office furniture, photographic equipment, firearms, pens and pencils, costume jewelry, office equipment, etc. Some electroplating process with unique usefulness includes chodium plating for reflection, silver and chodium plating for electrical properties and gold plating for god electrical contact. One of the most important surface effects is increasing solderability by plating with tin. For a plated coating to perform its function, it must adhere to the substrate.

2.8.2 PROTECTION AGAINST CORROSION

Electroplating of metal surfaces gives good protection against mositure, humidity, acid fumes and perspieration. The final criterion resistance is actual service, but accelerated test are needed both for projecting device life and for acceptance or rejection in production. No accelerated corrosion test can exactly duplicate the corrosion process that takes place during the life of the part while merely spreading up. Although, the well known neutral salt spray (fog) test (ASTMB 117) is widely used and specified, it is not a good measure of the corrosion resistance of plated coating. It may however, be useful as a quality control systems.

2.8.3 ENGINEERING OR MECHANICAL PROPERTIES

Situations where electroplates are applied for their mechanical, physical, or chemical properties rather than primarily to protect or justify the substrate reflects there engineering applications. Metals in this connection includes chromium, iron, copper, lead, silver, gold and tin.

The mechanical properties of nickel, that is, hardness, tensile strength, and stress can be varied by controlling the conditions of deposition. These properties,

4.0 RESULT, DISCUSSION OF RESULT AND CALCULATION

RESULTS

EXPERIMENT 1 DATA

Time (min)	0	1	2	3	4	5	10	15	20	25	30	35	40	45
Voltage (Volts)	109.5	109.5	111.0	114.0	114.0	114.0	114.0	111.0	111.0	111.0	111.0	111.0	111.0	111.0

EXPERIMENT 2: DATA

Time Min	0	1	2	3	4	5	10	15	20	25	30	35	40	45	50	55	60
volts	111.0	111.0	111.0	111.0	111.1	111.6	112.9	113.5	113.4	113.4	113.4	113.7	113.7	114.6	114.9	113.7	115.5

EXPERIMENT 3 DATA

Time (mins)	0	1	2	3	4	5	10	15	20	25	30	35	40	45
voltage (v)	109.5	110.4	110.4	110.4	110.7	110.0	107.4	107.4	107.7	107.7	107.4	107.4	107.7	107.7

EXPERIMENT 4 DATA

time (mins)	0	1	2	3	4	5	10	15	20	25	30	35	40	45
voltage (v)	104.4	104.4	106.5	107.4	107.4	107.1	106.5	106.5	107.1	108.0	105.9	110.4	111.0	111.3

EXPERIMENT 5 DATA

Time(mins)	0	1	2	3	4	5	10	15	20	25	30	35	40	45
voltage(v)	105.0	105.0	105.9	106.3	107.7	107.7	105.9	107.7	107.7	107.7	106.8	105.0	107.7	107.7

Table* COATING THICKNESS AFTER ELECTROPLATING

Material buckles	Weight before plating (g)	Weight after plating(g)	Coating thickness
1	3.90	3.95	0.05
2	3.91	3.96	0.05
3	3.89	3.97	0.08
4	3.91	3.98	0.07
5	3.90	3.98	0.08
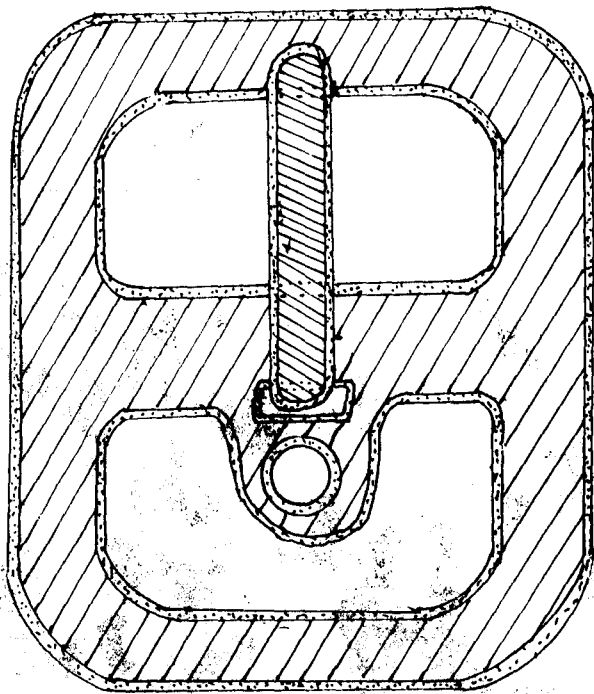
(b) AFTER COATING
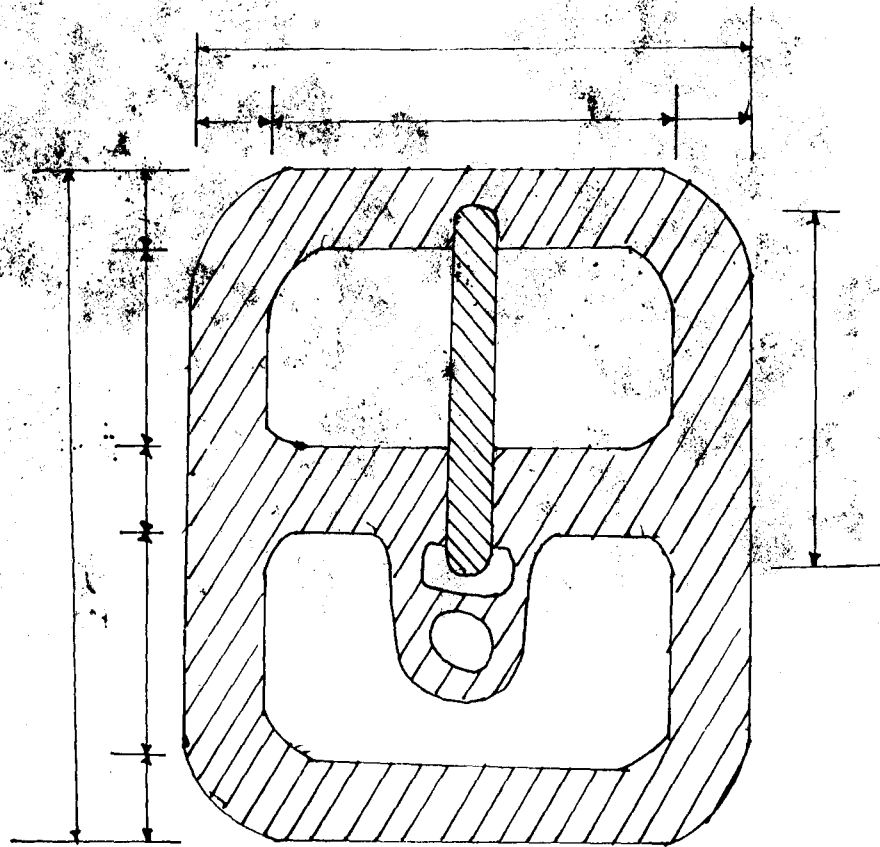
(a) BEFORE COATING

FIGURE 2 : SECTIONAL VIEW OF THE BUCKLE

SCALE : 1:2

BARREL

3mm ⌀
ERFORATION

COPPER HOOKS
TO CATHODE

CATHODE
CONTACT AREAS

INSULATED CABLE TO
HOOKS & CATHODE CONTACT

GEAR

FIGURE 4
PLATING BARREL

THICK RUBBER
LINNING (2.5mm)

200mm

200 mm

400 mm
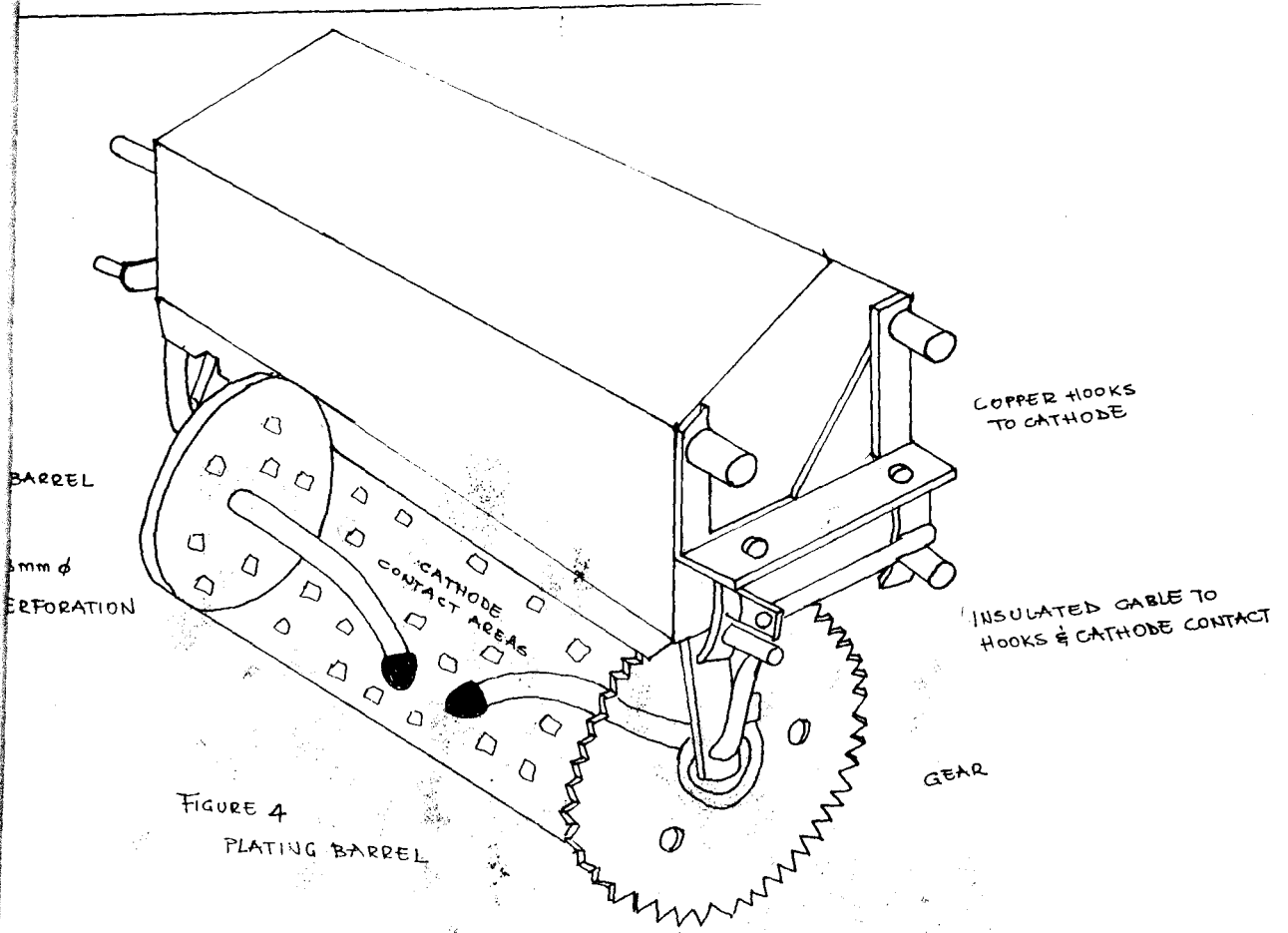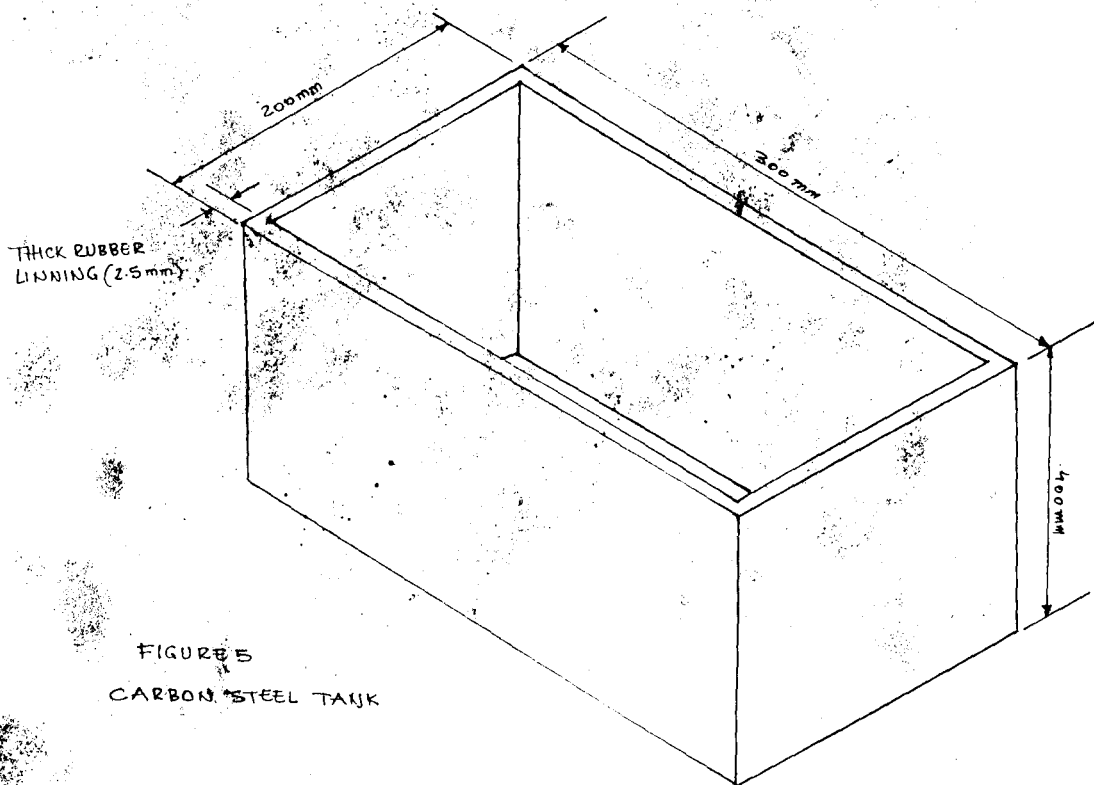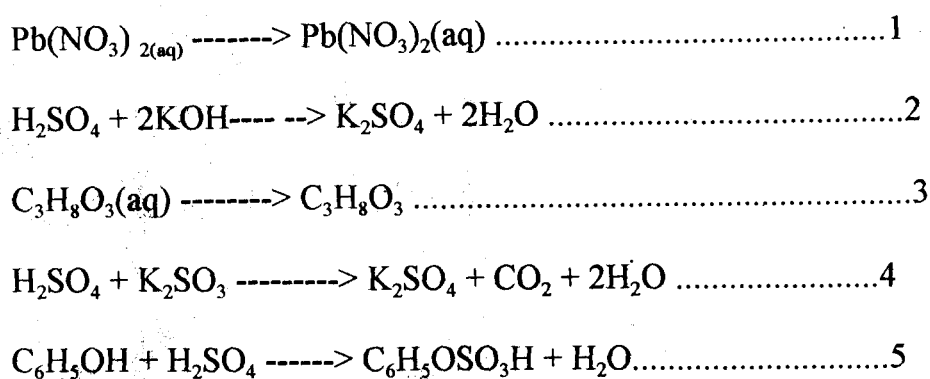
FIGURE 5
CARBON STEEL TANK

CHAPTER FIVE

DESIGN OF ELECTROLYTIC CELL THAT

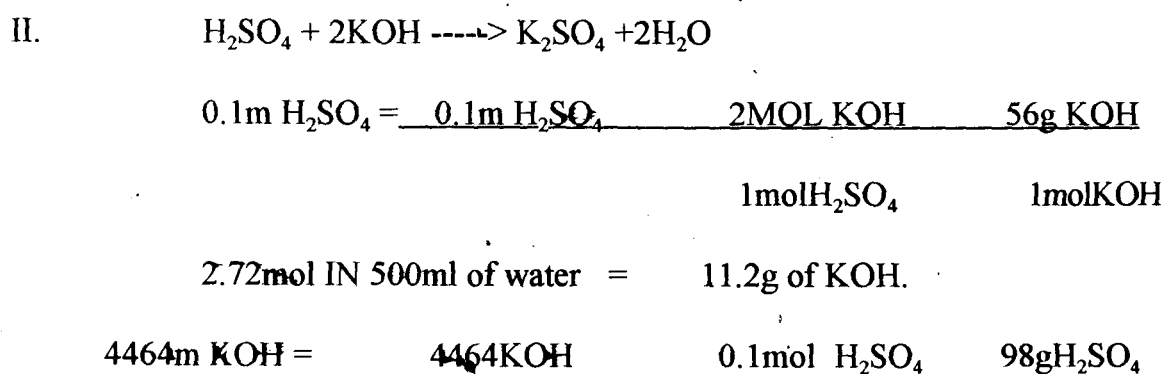# CAN ELECTROPLATE 300 SHOE BUCKLES

5.1   EQUIPMENT AND DESIGN

Thereaction leading to information of the electrolytic bath can be described by

the following equations of reaction

$$Pb(NO_3)_{2(aq)} \text{-------> } Pb(NO_3)_2(aq) \text{ ....1}$$

$$H_2SO_4 + 2KOH \text{------> } K_2SO_4 + 2H_2O \text{ ....2}$$

$$C_3H_8O_3(aq) \text{--------> } C_3H_8O_3 \text{ ....3}$$

$$H_2SO_4 + K_2SO_3 \text{---------> } K_2SO_4 + CO_2 + 2H_2O \text{ ....4}$$

$$C_6H_5OH + H_2SO_4 \text{------> } C_6H_5OSO_3H + H_2O \text{............................5}$$

BASIS 1 LITER SOLUTIOON

MATERIALS	CONC. / LITER	MOLE WT.	MOLARITY
$Pb(NO_3)_2$	97g/liter	331g	0.2931
K_2CO_3	35g/liter	138g	0.254
KOH	250g/liter	56g	4.464m
PHENOL	2g/liter	24g	0.0231
H_2SO_4	272ml in 500m/H2O =4.99g/liter	98g	0.1m
GLYCERINE	65ml/liter = 81.97g	92g	0.891m

Total = 470.96

II. $H_2SO_4 + 2KOH \text{----> } K_2SO_4 + 2H_2O$

0.1m H_2SO_4 = __0.1m H_2SO_4_____   2MOL KOH_____   56g KOH

                                                        1molH_2SO_4 1molKOH

      2.72mol IN 500ml of water   =   11.2g of KOH.

4464m KOH =   4464KOH   0.1mol H_2SO_4 98gH_2SO_4

Amount of H$_2$SO$_4$ reacted = 43.75g

Excess H$_2$SO$_4$ unreacted = (43.75 - 4.99)

$\qquad$ = 38.76g

Limiting reactant of KOH = 250g/liter

5.1.1 K$_2$SO$_4$ produced

2molKOH = 1molK$_2$SO$_4$

4.46mol KOH = ½ * 4.464 = 2.232 K$_2$SO$_4$

Molar mass of K$_2$SO$_4$ = 174g

Concentration = 2.232 * 174 = 388.368 K$_2$SO$_4$ produced

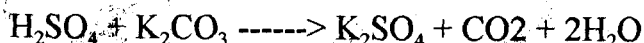### 5.1.2 Water produced

2molKOH = 2molH$_2$O

4.464 KOH = (2/2) * 4.464 = 4.464mol OF WATER

Mass of H$_2$O produced = 4.464 * 18 = 80.325g

$\qquad$ = 80.325cm^3 of water

Considering second equation

H$_2$SO$_4$ + K$_2$CO$_3$ ------> K$_2$SO$_4$ + CO2 + 2H$_2$O

Concentration of excess H2SO4 in mixture = 38.76g

Molarity = 38.76 / 98 = 0.3955mol K$_2$SO$_4$

0.3955molH2SO4 = 0.3955molH$_2$SO$_4$ $\qquad$ 1molK$_2$CO$_3$ $\qquad$ 138gK$_2$CO$_3$

$\qquad\qquad\qquad\qquad\qquad$ 1molH$_2$SO$_4$ $\qquad$ 1mol H$_2$SO$_4$

$\qquad$ = $\qquad$ 54.579gK2CO3

0.254mol K$_2$CO$_3$ = $\qquad$ 0.254molK$_2$CO$_3$ $\quad$ 0.5molH$_2$SO$_4$ $\qquad$ 97g H$_2$SO$_4$

$\qquad\qquad\qquad\qquad\qquad$ 1molK$_2$CO$_3$ $\qquad$ 1mol H SO

$\qquad$ = $\qquad$ 12.446gH$_2$SO$_4$

Amount of H$_2$SO$_4$ reacted = 12.446g

Excess H$_2$SO$_4$ = 38.76 - 12.446 $\quad$ = 26.314g

= 0.114 * 4184 J/gK = 0.4757J/Jk

TABLE 5: CALCULATION OF SPECIFIC HEAT OF K$_2$CO$_3$

ELEMENTS	Molecular weight	Heat capacity
K	78g	6.938 * 2 = 13.876
C	12g	2.225 * 1= 2.225
O	48g	4.074*3 = 12.222
TOTAL	138g	28.325C/deg.

Cp = 28.323 / 138 = 0.20523C/gK

So therefore, 0.20523 * 4.184 = 0.8587205J/gK

Cp 2K$_2$CO$_3$ = 0.8587205 * 2 = 1.717441j/gk

TABLE 6: CALCULATION OF SPECIFIC HEAT OF KOH

ELEMENTS	Molecualr weight	heat capacity
K	39	6.9381*1= 6.9381
O	16	4.074 * 1 = 4.074
H	1	4.970 * 1 = 4.970
TOTAL	56	15.982C/degM

Cp = 15.982 / 56 = 0.2853928C/jk

= 0.28539 * 4.184

= 1.1940837

Cp 2KOH = 1.194084 * 2

= 2.38817J/gk

TABLE 7: CALCULATION OF SPECIFIC OF H_2SO_4

ELEMENTS	Molecualr weight	Heat capacity
H	1	4.970 * 2 = 9.94
S	32	5.7264 * 1= 5.7264
O	16	4.074 * 4 = 16.296
TOTAL	49g	31.9624

Cp = 31.9624 / 49

= 0.6522938

Cp H2SO4 = 0.6522938 * 4.184

= 2.729198J/gm

TABLE 8;

ELEMENTS	Molacular Weight	HEAT CAPACITY
C	36	2.225 * 3 = 6.675
H	8	4.97 *8 = 39.76
O	16	4.074 * 3 = 12.22
TOTAL	60Kg	58.657Cal deg.mol

Cp = 58.657 / 60 = 0.97765C/g mol.

Cp = C3H8O = 0.97765 * 4.148

= 4.090486J/gmol.

TABLE 9:   CALCULATION OF SPECIFIC HEAT OF C_8H_5OH

ELEMENTS	Molecular Weight	Heat capacity
C	36	2.225 * 6 = 13.35
H	6	4.97 * 5= 24.85
O	16	4.07 *1 = 4.07
TOTAL	58g	42.274Cal. / g.Mol

Cp = 42.274 / 58

  = 0.72886 C/g.mol

Cp C_6H_5OH = 0.72886 * 4.184

  = 3.049559 J/gmol.

TABLE 10: CALCULATION SPECIFIC HEAT OF K2SO4

ELEMENTS	Molecualr Weight	Heat Capacity
C	12	2.299 * 1= 2.299
O	32	4.088 * 2 = 8.164
TOTAL	44	10.463C/gmol.

Cp = 10.463 / 44

  = 0.238C/gk

Cp1 CO2 = 0.238 * 4.184 = 0.996J/gK

Cp 2 CO2 = 2 * 0.996 = 1.992J/gK

Σcpt1 = Cp reactant $Pb(NO_3)_2$+Cp reactant $2K_2CO_3$ +Cp reactant C_6H_5OH

Σcpt1 = 0.4758 + 4.0904876 + 2.729198 + 2.38817 + 1.717441 + 3.049559

  = 14.450556J/gK

Σcpt2 = cppdt $Pb(NO_3)_2$ + Cpp dt + $C3H_8O_3$ + Cppdt 2K2SO$_4$ + Cpdt $2H_2O$

+ CP2CO$_2$

ΣcpT2 = 0.4757 + 4.0904 + 1.730175 + 25.200 + 1.992

  = 33.488363

Δcp = Σcppdt - Σcp reactant

5.15 COST OF BARREL PERFORATIONS

Drilling of 5mm hole =50k

Drilling of 200 holes=200*50k=N100.00

5.16 COST OF ELECTRIC MOTOR.

0.5HP electric motor=N8000.00

5.17 COST OF COPPER RODS

cost of 0.5m x 0.15m =N150.00

cost of 3rods =N150 x3 =N450.00

5.18 COST OF CABLE

3.0mmcable of 1.5m =N300.00

5.19 COST OF COPPERHOOKS

1hook cost N30.00

2hooks cost =N30 x2 =N60.00

5.20 COST PROPYLENE BARS

cost of thick propylene = 0.2m*0.02m*0.01m =N100.00

Cost of 2 =N100 x2 =N200.00

5.21 COST OF GEAR (POLY -PROPYLENE)

1 gear of diameter 25mm =N150.00

2 gear of same diameter= N150.00 x2=N300.00

5.22 COST OF FLEXIBLE CATHODE CONTACTS

A pair of flexible cathode contact of length 120mm and diameter 5mmcost N250

5.23 COST OF ANODE

300g of pure lead cost N300

5.24 COST OF THE BUCKLES

1 brass shoe buckle of surface area 0.157dm^2cost =N2O

5shoe buckles cost =N20x 5 =N 100

Total cost of major item of equipments N12,521.48K

5.25 COST OF RAW MATERIALS

pb(N03)2

500g of pb(NO3)2 cost N800.00

4000g =4000 x800/500 = N6400.00

K2CO3

500g of it cost N1200

1500g =1500x1200/500 =N3600.00

KOH

500g of it cost N900.00

1200g =1200x 900/500 =N 2160.00

H2SO4

500ml of it cost N500.00

2000ml =2000x 500 /500 =N2000.00

DISTILLED WATER

1 litre of water cost N10.00

100litres =N100x 10 =N1000.00

GLYCERINE

1litre of it cost N15

100 litre of it = N15 x100=N 1500.00

PHENOL

10litre of it cost N25

800litre =800x 25 /10 =N2000.00

Total cost of raw material =N18660.00

COST OF ENERGY

Total energy required for the process $= 9500 \; 10^6$ J

If 1KWh electricity utilises 3.6 x10^6J

- Then 9500 x10^6J will require =9500 x10^6 /3.6 x10^6

$= 2.639$ Kwh

if 1kwh cost 3k

2.639kwh =2.639x 3 =N 7.92K

so,total cost of energy =N7.34K in 10 mins of operation

5.26 PROCESS CONSIDERATION OF OPTIMIZATION

It is advisable to electrolyte of high conductivity ie having sufficient ionic content plating solutions .This will minimise the power consumption toohmic polarization in the solution and also increasing the throwing power ofthe bath

2. Agitation is recommended to permit plating rate hence reducing the time of plating

3. Itis also advisable to operate at the maximum current density in order to deposit on the cathode substrate at the shortest possible time

4 A high temperature gives rise to useful resultsdue to increase in salt solubility , thus permitting greater metal and higher current density.

5.27 SAFETY AND HEALTH IN ELECTROPLATING

Each stages of electroplating process is eminent to different types of dangers due to the us e of toxic ,concentrated solutions and electricity in addition their is possible exposure to dust from work material and gases and vapour of the organic solvent used in the cleaning process

Health and safety in electroplating industry involves

1. Atleast a member of the staffs should be trained on first aid precautions and application

2. During polishing ,mixing of the salt, undertaking ,pickling ,plating processes quality control and effluent treatment ,rubber aprons ,overall wears ,hand gloves should be worn at all times.

3. Maitenance of the equipments used in carryingout the process is essential .This willreduce the effect of contamination .

Allworking staffs in the workshop should ensure that appropriate dresses ,tools and

$$= 33.48836 - 14.45056$$

$$= 19.0378 \text{J/kgK}$$

$$Q = M_{min} \Delta cp \, (t2 - t1)$$

$$T1 = 306k; \quad T2 = 313k$$

$$\Delta cp = 19.0378 \text{J/kgK}$$

$$M_{max} = 43476. \, 179g$$

Heat of process $\quad Q_H = M\delta cp \, (T2 - T1)$

$$= 43476.179 * 19.0378 \, (313 - 306)$$

$$= 827691. \, 1 * 7$$

$$= 5793837.7J$$

$$= 8.276MJ.$$

5.7 ELECTRICAL ENERGY BALANCE FOR THE BARREL GEAR

Power required $= (745.7w + 1.8642w) \, / \, 1Hp$

$$Q_{HE} = VIT = P*T$$

$$= 1.8642 * 2700$$

$$= 5033.34J\backslash$$

5.8 ELECTRICAL ENERGY REQUIRED FOR ELECTRODEPOSITION

BASIS

$$1 = 162 \, ; \quad T= 45 \qquad \text{min.; and } V = 3.70 \text{Volt}$$

$$Q_D = VIT$$

$$= 162 * 3.70 * 2700$$

$$= 1618380$$

Total energy balance for overall process

$$Q_t = Q_H + Q_E + Q_D$$

$$Q_T = 5793837.7 + 5033.34 + 1618380$$

$$= 8496171J$$

$$= 8.4962MJ$$

5.9.0 DISCUSSION OF RESULT

5.9.1 GENERAL OVERVIEW OF ELECTROPLATING PROCESS

I n the electroplating process the movement of Pb ions from anode to the cathode was observed. This is due to the electrochemical effort of process. Also become darker due to the decomposition of Pb as a result of the dessipation of electricity as heat. This keeps fluctuating for the fact concentration of Pb in solution keeps on changing.

5.10 THE DEPENDENCE OF TIME OF THE ELECTROPLATING PROCESS

For particular electrolyte used for electroplating, there is an uptimum time required for effect decomposition of the metal (Pb) on the substrate (buckle). Although the amount of Pb deposited on the buckle is a function of time, but from experimental result it was observed that at 45minutes the best was got. So in the essence the uptimum time is 45 minutes. When the time was increase further, although there are more clusters of Pb on the buckle but the binding power of the lead is not as shining of that it was stored at 45 minutes.

5.11 EFFECT OF THE ADDITIVES ON THE ELECTROPLATING PROCESS

There are four main additives used, they are needed to enhance the binding power of the lead on the bukcle oand also to give shining colour.

It was obsreved that where KOH was put in exces the overall desired property desired,that is, high adherance and shining colour it was noted that when phenol was put in excess, it usually leads to the depreciation of the deposited Pb. So a small quatity of phenol as additive is desired. The gycerine is added in small quantity due to the fact that it is just added to smoothe the surface of the electroplated buckle. A little addition of K_2CO_3 changes the colour of the electrolyte to the white which is desirable.

5.12 OPTIMUM CONDITION FOR THE ELECTROPLATING PROCESS

By the thorough evaluation of all the parameters that contribute to the effective electroplating process, it is obsreved that the electroplating time should be at 45 minutes and the additive should added in relatively small qautitty as compared to the amount of $Pb(NO_3)_2$ added.

5.13 EVALUATION OF ELECTROLYTIC CELL DESIGN

The cell consists of stainless steel tank incoporated with another band to serve as an insulator. The cost of construction was estimated to be ₦11,436.50. So, in essence, the setting up of an electroplating industry is relatively cheap and profitable owing to the low cost of construction equipment.

materials be used during the process

5. In unavoidable cases where worker becomes avictim of any of the safety precautions, first aid treatment should be imlemented quickly depending on the kind of defect before taking to the clinic .

5.14   COST OF MAJOR ITEMSOF EQUIPMENT

SPECIFICATION

5.14.1BARREL

1. Size

Length = 120mm

Diameter=90mm

2. Optimum plating surface area m2=075m2.

3. Typic

al current amperes=80 amps

5.14.2 DETAILS

1. Size

Length = 300mm

width=350mm

Height= 400mm

Drive 5.14.3T =0.5HP gear motor

5.14.3 TANK (CARBON STEEL)

Thickness of the metal sheet =2.5mm

cost of 1*1m =N1500

5.14.4 TOTAL AREA OF THE SHEET

2 Face of length*breadth =2(0.300*0.20m) =0.12m2

2 face of height*width= 2(0.400 *0.350m) =0.28m2

1 face of length*width =1(0.300*0.350m) =0.105m2

Total area of sheet =0.505m2

If 1m 2

Then 0.505 m2 will cost N1500*o.505 =N757.50

5.14.6 RUBBERLINING

Area of the rubber lining =Aera of thetank=0.505m2

1m2 rubber cost =N230

so,0.505m2 rubber =230*0.505 =N116.15

BARREL

Total surface area ofbarrel = DL +πD2/2

Length = 0.18m

Diameter=0.14m

Barrel area =2.142*0.14*0.18+$\dfrac{3.142*0.14*0.14^2}{2}$

= 0.079+0.03079
=0.1098m2
1m2polypropylene material cost N800
0.1098will cost = 0.1098*N800
=N87.83

CHAPTER SIX

6.0   CONCLUSION AND RECOMMENDATION

6.1   CONCLUSION

Based on the result got in the experiment, the following conclusion are made,

(I) Optimum time required for the electroplating of buckle in the lead is 45 minutes.

(ii) For the effective eletroplating , small amount of KOH, K_2CO_3, glycerine and phenol should be added relative to the amount of $Pb(NO_3)_2$.

(iii) The construction of the electrolyte cell is cost effective as regard to the low cost of construction.

6.2   RECOMMENDATIONS

The following recommendations are made to further enhance the effective electroplating process;

i. The chemical used should be fresh, that is, should be prepared fresh before use.

ii. The industry should be located where there is stable current to enhance uniform deposition of the lead on the buckle.

iii. The industry should be situated very close to the market.

1.1 CALCULATION OF SURFACE AREA OF THE BUCKLE

From figure 2

Area of shaded portion

Total area of rectangle = 3.3*3.4 =11.22 cm^2

Area of (3)   = 0.25 *1.6 =0.40 cm^2

Area of (1)   = 1* 2.1   = 2.1cm^2

Area of circle   = π *0.4^2 -π *0.15^2

                       = 0.50265 -0.0707 =0.432 cm^2

Area of the hole   = 1*2.1 -0.432 =2.1-0.432=1.668cm^2

Area of the shaded portion = 11.22+0.4 -1.668- 2.1=7.85 cm^2

Total surface area   = 2*7.85 =15.70cm^2 =0.157dm^2

CALCULATION OF CURRENT INTENSITY

I=Dc *A

Where I =current in amperes

Dc=cathode current density A/dm^2

A_k=area of the buckles in m^2

I=300*0.00157=o.47amp

APPENDICE B

CALCULATION OF THE COATING THICKNESS

$\delta = \tau Dc\dot{\eta}/22.3$

Where

δ = coating thickness in μm

Dc = cathode current density in A/m^2

τ = electrolytic timein hour

ratio of the lead density(11.4g/m^3)to its electrochemicalequivalent

(106195g/a.h)

1.2.1 CALCULATIONOF THE CURRENT($\dot{\eta}$)

$\dot{\eta} = G/Gth$

Gth = IτE

G = actual amount of substance in electrolysis

Gth = Theoritical amount calculated from faraday's law at the same electric power

E = electrochemical equivalentof buckles

1 1.2.1. E for buckles

E for brass = Brass constit40% Zn and 60%copper

Efor zn =.0.000340

E for cu =3.29*10-7g/Ah

E forbrass=40/100*zn+60/100*3.29*in 3.29*10-7

= 1.36*0.00034 +1.974*10-7=1.361974*10-4g/Ah

1.2.1.2

$\dot{\eta}$1 for buckle with initial massof 3.90 at 45 mins

$\dot{\eta} = \dfrac{3.95\text{-}3.90}{0.5*0.75*1.361974*10\text{-}4}$

=9.789
1.2.1.3

$\dot{\eta}$2 for buckle with initial mass of 3 .91g at 60mins

$\underline{3.96\text{-}3.91}$
0.5*1*1.361974*10-4
=7.34

1.2.1.4

η̇3 for buckle with mass of 3.89g at 45 mins

η̇3=$\underline{3.97\text{-}3.89}$
      0.5*0.75*1.361974*10-4
=15.66

1.2.1.5

η̇4=$\underline{3.98\text{-}3.91}$
      0.5*0.75*1.361974*10-4
=13.7

1.2.1.6
η̇5 for buckle with initial mass of 3.90g at 45 mins
η̇5=$\underline{3.98\text{-}3.90}$
      0.5*0.75*1.361974*10-4
=15.66

1.3 CALCULATION OF INDIVIDUAL COATING THICKNESS

δ1=$\underline{0.75*300*9.789}$
      1.0735*10^-3
=2.052μm

δ2=$\underline{0.75*300*7.342}$
      1.0735*10^-3
=1.5μm

δ3=$\underline{0.75*300*15.664}$
      1.0735*10^-3
=3.28μm

δ4=$\underline{0.75*300*13.705}$
      1.0735*10^-3
=2.87μm
δ5=$\underline{0.75*300*15.664}$
      1.0735*10^-3
=3.28μm

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