



PARAMETERS AFFECTING QUALITATIVE AND EFFICIENT ELECTRODEPOSITION OF METALS IN AN ELECTROLYTIC CELL: A REVIEW

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ABSTRACT

Electroplating has been a useful practice in the laboratory and industry for a long time. Its usefulness is more profound in oil industries for corrosion prevention and control, automobile industry, jewelries and decoration. Today, the scope of electroplating has expanded considerably with many players exploring its advantages and optimizing its parameters for enhanced productivity. The paper presented a review of electroplating with the aim of making concise information available on its process parameters and its process optimization. The review was compiled from several major work which pertained to parameters affecting qualitative and efficient electrodeposition of metals in an electrolytic cell. Results gathered included actual effect of identified parameters and interplay of parameter on quality of electrodeposition and microstructure of deposited metal. Important recommendations were made to further enhance the practice of electrodeposition.

Keywords: Electroplating, Efficient Electrodeposition, Electrodes, Electrolyte, Operating Parameters

INTRODUCTION

Electrodeposition is a technological process for the production of a material using an applied potential difference or current flowing in an electrolytic cell containing metal ions to form metal, metal alloys or composites as a coating for another metal (Zangari, 2018). The product of electrodeposition is a dense, uniform and an adherent coating usually of metal on another conducting surface (ASTMB374-06, 2019). This process is usually carried out either to achieve properties not originally exhibited by a material or to enhance such properties such as wear resistance, corrosion protection, lubricity, conductivity, aesthetics, etc. Electrodeposition is an important industrial process; this technology has a long history dated back to eighteenth century. In 1801, electroplating was reported and experiments for depositing dendritic metallic lead and copper onto a surface using Volta piles (Garcia & Burleigh, 2013). Also, an Italian scientist, Luigi Brugnatelli, in 1805 published on the use of Volta piles to deposit a layer of gold onto another metallic surface (Thomson, 1813).

Electrodeposition has equally found an important use in the area of microfabrication and nanotechnology research (Andre & Maximiliano, 2006). It is therefore of important proportion to be able to obtain a good quality metal deposition and through an efficient process. Many authors have researched the field of electroplating with impressive findings and report. The special

interest in the field is connected with the multifarious applications of electroplating in research and industry. Electroplating has many advantages in the industry and these constitute a major drive for research. The electrodeposition of metallic, ceramic or polymeric particles embedded in a metal matrix has become a mature technology which continues to evolve and diversify (Walsh *et al.*, 2020). The present study provides a concise review of electrodeposition parameters and their interplay during the process.

BACKGROUND

Electrodeposition or electroplating as electrochemical process obeys electrochemistry fundamental laws, Michael Faraday formulated two laws of electrolysis indicating the balance of mass and charge in electrochemical reactions (Zangari, 2018). Lou and Huang held that whenever a current is passed through an electrolyte, a chemical reaction takes place at the electrodes (Lou & Huang, 2006). Metal deposition takes place according to Faraday's laws of electrolysis (Walsh *et al.*, 2020). The reaction is as depicted by the following equations;

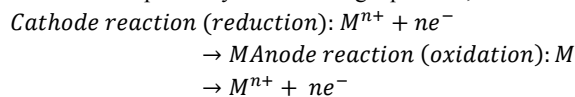


Figure 1 shows the schematics of an electrolytic cell for deposition of a metal from a solution of the metal salt.

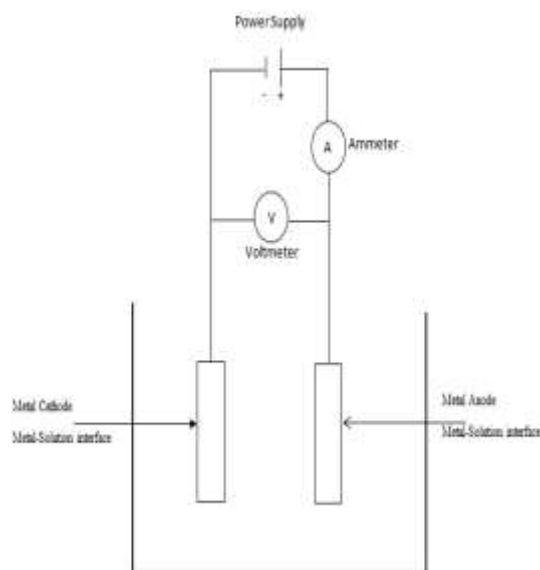


Fig. 1: Schematics of an electrolytic cell for plating metal "M" from a solution of the metal salt "MA"

Faraday's first law is summarized by the equation;

$$M = KQ = KIt \quad (1)$$

where M is mass deposited at cathode in grams (g), K is the Proportionality constant – electrochemical equivalent (mg/As), I is Current in amperes (A) and t is Time for which current flows in seconds (s). Given a negligible secondary reaction at the electrodes and a constant current flow, the above equation will accurately estimate mass deposited (Andre & Maximiliano, 2006). Quality electrodeposition depends on right combination of process or operational parameters. Notable emphasis has been on investigation of process parameters as it affects electrodeposition of metals from an electroplating bath (Tuaweri *et al.*, 2013; Bograchev & Davydov, 2019). However, for a transient current condition where the instantaneous current is not constant, the weight deposited and the thickness of deposition can be estimated by relating equations (2) to (5) below to derive equation (6)

$$m = \frac{Q}{nF} \quad (2)$$

$$Q = \int Idt, \quad (3)$$

$$K = \frac{M_w}{nF} \quad (4)$$

$$w = \frac{M_w}{nF} \int Idt \quad (5)$$

$$\delta = \frac{W}{\rho A} = \frac{M_w}{nF\rho A} \int Idt \quad (6)$$

and by solving the resulting equation (6) (Han, 2012). Where ρ is the density of the metal (g/cm^3), N is the number of valence

electron in the metal being deposited, A is the area of deposition (cm^2), F is the Faraday constant which is $9.65 \times 10^4 (\text{C/mol})$, δ is the deposition thickness in cm, M_w is the atomic weight of the metal being deposited and W is the weight of the deposit. The accuracy or otherwise of these equations is impacted by other factors that generally impact electrodeposition such process factors were listed to include bath concentration, agitation, time, current density, pH and electrolyte temperature (Kumar *et al.*, 2015).

These and others are the factors that inter-relate to influence the quality of deposit (Sadiku-Agboola *et al.*, 2011). Also of importance is the influence of process parameters on electrodes used in electroplating (Begum *et al.*, 2013). Of course, this essentially affects the anode. A consideration of control and optimization of identified process parameters is significant for successful electroplating (Prasad *et al.*, 2000; Noh, *et al.*, 2011; Khedekar *et al.*, 2016).

The microstructure of electrodeposit is often a good pointer to quality electrodeposition (Trzaska & Trzaska, 2008). In general, a smooth morphology, small grain size or refined grains, and fine texture are desirable (Rashidi & Amadeh, 2010; Esmar, *et al.*, 2020). Such structures are generally observable using electron microscopes.

ELECTRODEPOSITION PARAMETERS

A number of parameters interplay during electrodeposition and may be broadly categorized into chemical, electrical and physical parameters. These parameters are briefly considered in the following paragraphs.

CHEMICAL PARAMETERS

The electrolyte is a major component in electrodeposition and its composition determines the integrity of the plated workpiece and the process. Chemical parameters revolve centrally around the electrolyte or bath and pertains to concentration of the electrolyte, pH, and additives among others.

Concentration of Electrolyte

Prasad *et al.*, (2000) found that although bath with 0.20M cobalt sulphate yielded acceptable deposition, increasing the concentration to 0.40M resulted in enhanced current efficiency during deposition of boron-containing amorphous metallic layer of cobalt-molybdenum alloy. High bath concentration will ordinarily increase the deposition rate of the plating process (Kumar *et al.*, 2015). To obtain a Cu-coating on Magnesium – Lithium(Mg–Li) alloy substrate, Yin *et al.* found a 50 g/L concentration of the main salt to be optimum for the electrolyte (Yin *et al.*, 2013). The surface morphology and composition of the electrodeposit in Tin-Bismuth (Sn-Bi) deposition is influenced by concentration of Tin (Sn) metal salt in the electrolyte. Sn content in the deposits increased with an increase of Sn metal salt content in the bath (Heonget *et al.*, 2012).

It was reported by (Sadiku-Agboola *et al.*, 2011) that in bright nickel plating, the concentration of nickel salt has an effect on the weight of nickel deposited on the cathode. Agasti *et al.*, (2014) while studying optimization of zinc salt concentration in

co-electrodeposited Copper Zinc Tin Sulfide ($\text{Cu}_2\text{ZnSnS}_4$) also reported that Zn content (atomic %) becomes very high for the films with ZnSO_4 bath concentration 0.06M and more compared with concentration of 0.03M. During composite electroplating of Zinc Nano- Titanium dioxide, the weight percentage of Titanium dioxide (TiO_2) particles embedded in the deposited composite were influenced by the concentration of TiO_2 in the electroplating bath among other factors (Mokabber *et al.*, 2013). The influence of electrolyte concentration on electrodeposition was underscored by Yu, *et al* in their work, they showed that the effect of concentration may be complex depending on other interacting conditions by concluding that Iron (Fe) content of the deposited Nickel Iron Tungsten (NiFeW) alloy increases from 4.37 to 8.1% with the increasing of Iron (II) chloride (FeCl_2) concentration in the range of 1.5–3.6 gl^{-1} and it tends to increase very slowly with higher FeCl_2 (Yuet *et al.*, 2017). Analysis on the influential factors of Copper (II) Cu^{2+} electrodeposition revealed that concentration of Cu^{2+} in the electrolyte influenced amount of Copper deposited at cathode (Sui *et al.*, 2017). Metal salt concentration is a crucial and influential factor in electrodeposition as myriads of work done by researchers pointed in this direction.

Use of Additives

Presence of additive tends to increase current efficiency and soundness of electrodeposits, for example Presence of an additive 17.0g/L $(\text{NH}_4)_2\text{SO}_4$ to the electrolyte in the electrodeposition of Cobalt Boron Molybdenum (CoMoB) amorphous alloys increased the soundness of the deposits (Prasad *et al.*, 2000). Tang reported an improvement in ultimate tensile strength as well as ductility of Nickel plated from electrolyte with presence of additive compared with bulk Nickel (Tang *et al.*, 2010). Certain surface properties such as hardness may be increased by additives (Trzaska & Trzaska, 2008). The use of appropriate additives and in the right concentration can be used to increase growth of deposits during electrodeposition (Si *et al.*, 2016). Some additives are used with the primary purpose of improving the physical appearance of the plated metal as depicted by the electrodeposition of a bright cyclic multilayer Zinc cobalt (Zn-Co) from electrolyte having a stable bath with Thiamine Hydrochloride (THC) as additive (Bhat & Hegde, 2012).

Electrolyte pH

According to (Prasad *et al.*, 2000), the best electrodeposition results are obtainable in the pH values ranging from 5.0 to 6.0. Increase in pH results in increase in cathode current efficiency (Tuaweri *et al.*, 2013). Additives are often added to the electrolyte but the pH of the bath determines adsorption of such additives (Kumar *et al.*, 2015). Furthermore, Kumar & Clement, (2011) claimed that pH of the electrolyte contributes significantly to the variation in hardness of coatings. In the experiment to deposit Cobalt Copper (Co-Cu) alloy thin films on amorphous substrate by electrodeposition, Liu *et al.*, (2005) observed that a remarkably coarse surface at pH 2.2 gradually smoothed into much more refined structure at pH 5.05. pH also impacts on conductivity of electrolyte (Nia *et al.*, 2019).

The Faradic efficiency (FE), added mass and deposition rate

increased with increasing electrolyte pH, the FE, gained mass and deposition rate were the lowest at pH 2.0 but an increment of pH to 2.5 resulted in significant mass gain during a study of electrodeposition of Rhenium Iridium Nickel (Re-Ir-Ni) coatings (Wu *et al.*, 2014). In the work of Gomez *et al.*, (2003), different stages of electroplating of Copper Molybdenum (Co-Mo) was reported to be pH dependent. Selection of pH below 5 helps to reduce internal stress level of deposits, lower pH can be used to manage deposited material internal stress (Güler, 2016).

Throwing Power

Measure of an electrolyte's ability to deposit a uniformly on an irregular shaped cathode is referred to as the throwing power. Throwing power is impacted by current density, electrolytic conductance and agitation of the electrolyte (Lou & Huang, 2006). Higher throwing power is generally desirable in electrodeposition (Gamburga *et al.*, 2002).

ELECTRICAL PARAMETERS

Electroplating parameter that revolves around electric current in the electrolyte were reviewed in the following sections. Key electrical parameter reviewed include; current, current density, current distribution and current efficiency.

Electric Current

Flow of electric current is vital in electrodeposition, deposition takes place in an electrolytic cell when flow of electric current is established. Direct current plating does not produce a uniform deposition thickness when plating objects with corners, the outside corners end up with a thicker deposition compared with recessed corners (Lou & Huang, 2006). According to Chandrasekar and Pushpavanam, (2008), pulse current (PC) produces invariably fine-grained structures so that physical, chemical and mechanical properties are changed advantageously for many metals and alloys. It can equally affect the electro crystallization mechanism and control the physical and mechanical properties of the electrodeposit metal. The greater power consumption in PC can reduce bills for heating electrolytes but may cause overheating, particularly at low duty cycles. PC can influence the structure and composition of an alloy. Uniform composition can be obtained at high frequency pulses.

Periodic pulse reversed current waveform produces better uniformity, reduced porosity, improved hardness and decreased grain size of the deposited metal compared with direct current plating, it also allows precise control of the deposited material composition, crystallographic structure, texture and grain size (Trzaska & Trzaska, 2008).

Current Density

Current density is the ampere per unit area of the electrode (Lou & Huang, 2006) and it is inversely proportional to deposition time (Tuaweri *et al.*, 2013) and it determines the uniform coating of the final deposit (Kumar *et al.*, 2015). The higher the current density the higher the deposition rate up to a practical limit (Trzaska & Trzaska, 2008). A cathode current density of 50 mA/cm^2 was considered optimum by Prasad *et al* (2000). An increase in current density up to 75 mA/cm^2 results in a decrease in the average grain size and texture coefficient of nickel

coatings (Rashidi & Amadeh, 2010), this was corroborated by (Celis *et al.*, 2017) highlighting the dependency of grain size on current density and bath composition. Similarly, increase in the current density above 150 mA/cm² results in visible surface cracks in the coatings. The grain size and surface morphology of the coatings are also affected by change in current density (Oniku *et al.*, 2015).

Current Efficiency

Current efficiency is the percentage of electric current flowing in an electroplating bath that achieves the desired deposition results (Lou & Huang, 2006). It is expressed mathematically as;

$$\text{current efficiency} = 100 \times \frac{w_{Act}}{w_{Theo}} \quad (7)$$

where w_{Act} is actual weight of the deposited or dissolved material and w_{Theo} is the theoretical value of the deposited weight calculated according to equation (5). Changes in current efficiency of the bath appears to be insignificant over a range of current densities between 2.5-4.5 A/dm² after which current efficiency drops significantly (Tuaweri *et al.*, 2013).

Current Distribution

Current distribution is a complex phenomenon in electroplating, it is desirable to achieve a uniform current distribution in the electrolyte but this is usually concentrated around the edges of the electrodes and less around the middle (Tan & Lim, 2003). Uniform current distribution could be achieved by varying parameters such as agitation, bath conductance and electrolyte concentration individually as combining the parameter does not produce the expected cumulative effect (Tan & Lim, 2003).

Electrolytic Conductance

This is measurement of the ability of an electrolyte to conduct electricity and it is heavily dependent on the mobility of individual ions, viscosity and chemical composition of the electrolyte (Lou & Huang, 2006). Temperature increases conductivity of electrolyte (Kumar *et al.*, 2015). An electrodeposition simulation experiment performed with different electrolyte conductivities showed a decreasing thickness trend with decreasing electrolyte electrical conductivities (Mahapatro & Suggu, 2018).

PHYSICAL PARAMETERS

These are the third category of parameters affecting quality of electrodeposition. They include temperature, time, agitation, etc. these and others are described next.

Temperature of Electrolyte

Bogachev & Davydov, 2019 observed that the presence of temperature gradient enhances the uniformity of deposition as deposition happens at slower rates at lower temperatures. In effect, temperature affects conductivity of the solution. Increasing electrolyte temperature up to about 50°C results in corresponding increase in cathode current efficiency (Tuaweri *et al.*, 2013) but temperature of 45°C produces best result (Prasad *et al.*, 2000). Barbato *et al.*, (2008) opined that plating at a temperature of 50°C produces deposit with a better hardness and finer grain size.

Electrolyte pH and temperature changes seem to have some effect on the morphology and preferred orientation of the electrodeposits (Tuaweri *et al.*, 2013). Esmaret *et al.*, (2020) noted

that, high electrodeposition temperature in the vicinity of 45°C could produce smoother morphology and Ni grain refinement with attendant increase in coating hardness. Raising temperature encourages formation of coatings with coarse and porous grains (Kumar *et al.*, 2015; Ziti *et al.*, 2012).

Deposition Time

For a specific deposit thickness, deposition time is inversely proportional to current density (Tuaweri *et al.*, 2013) and it determines the plating thickness (Kumar *et al.*, 2015). Time has a direct effect on deposition thickness (Sadiku-Agboola *et al.*, 2011). Cvetković *et al.*, (2020) conducted electrodeposition of aluminium onto glassy carbon and aluminium substrates and found that the shape and size of Al grains deposited depended on the time of deposition and varied in size from nanometers to micrometers and in shape from regular crystal forms to needle-like and flake-like structures.

Study of optimization of process parameter for electrodeposition of Nickel Chromium (Ni-Cr) for coating variation shows that plating time have more effect on thickness variations (Khedekar *et al.*, 2016). Deposition time is directly proportional to the amount of metal coatings (Aygur & Üstünışık, 2009; Wahab *et al.*, 2013).

Agitation /Stirring of Electrolyte

Tuaweri *et al.* (2013) submitted that electrolyte agitation is slightly detrimental to the current efficiency after observing that bath agitation beyond 100 rpm reduced the current efficiency. Agitation is important for producing smooth coating and eliminating pits in coatings (Kumar *et al.*, 2015). Cathodic polarization studies showed that electrolyte agitation had significant effect on limiting current density and hence mass transport of the bath (Tuaweri *et al.*, 2013). Pits formation is eliminated by bath agitation and helps produce smooth coatings (Kumar *et al.*, 2015). Also, current distribution uniformity could be improved by optimal agitation of the electrolyte (Tan & Lim, 2003).

Agitation of electrolyte helps to replenish metal salt or its ions around the cathode, thins out the diffusion layer, minimizes gas bubbles capable of promoting pits formation and increases operating current density (Paunovic & Schlesinger, 1998).

Electrode Porosity

One of the main contributors for the high anode usage is anode porosity (Begum *et al.*, 2013).

Cathode Preparation

In order to achieve best electroplating results, workpieces are subjected to pretreatments such as cleaning, modification and rinsing. While cleaning deals with removal of surface contaminants which can be achieved using mechanical or chemical means such as degreasing, alkaline cleaning and acid pickling, (Lou & Huang, 2006) rinsing is done to remove drag-outs.

CONCLUSION

The subject of electroplating has been reviewed with a focus on operational parameters, their impacts and how they affect quality electrodeposition. It was observed that impact of each of the reviewed parameter is highly dependent on other parameters

interacting together during a given electrodeposition process. Cathode preparation is basic for electroplating, how well it is carried out has a direct effect on the final results. Electroplating is a very vast research field and these parameters are constantly being fine-tuned of to obtain a suitable plating environment for achieving desired result. Recommended optimum conditions varied being within limited scopes of discrete research endeavors. It is recognized that the field of electroplating will keep evolving with innovations for better product standard and optimum process design.

ACKNOWLEDGEMENT

The authors wish to thank Dr. Akinluwade K.J and Engr. Olabode O. for their invaluable contributions.

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