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Preparation and Characterization of Electrodeposited Nickel Hydroxide $[Ni(OH)_2]$ Thin Films

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Abstract: The present paper deals with the preparation and characterization of nickel hydroxide thin films using the potentiodynamic mode of electrodeposition from aqueous bath. During deposition scan rate, Bath concentration and number of potential cycle have been optimized. Also the various thin film characterization like Thickness of thin film was measured by using gravimetric weight difference method using sensitive microbalance, Structural properties of $Ni(OH_2)$ thin films were studied by XRD pattern, Surface morphological study using SEM technique, and surface wettability is studied using contact angle measurement.

Keywords: $Ni(OH_2)$, potentiodynamic mode of electrodeposition, Thickness of thin film, Structural properties of $Ni(OH_2)$, Surface morphological study, surface wettability

I. INTRODUCTION

With growing environmental concerns and increasing depletion of Petroleum-based fuels, many efforts have been concentrated on the development of alternative energy technologies. Super capacitors have been widely researched due to their interesting characteristics in term of high power density, excellent reversibility and cycling stability. The development of high performance electrode materials for super capacitors have attracted increased interests in recent years like conducting polymers, metal oxide etc. Amongst, low cost transition metal oxides have been exploited as electrode materials like Nickel hydroxide.
The present work is related to the supercapaticive behaviour of Nickel hydroxide films prepared by simple electro deposition method, which can offer the fulfilment of the requirements of the electrochemical super capacitors. Electro deposition is an isothermal process mainly controlled by electrical parameters, which are easily adjusted to control Film thickness, morphology, composition etc. Hence in the present work nickel hydroxide films are deposited by electro deposition i.e. potentiodynamic mode from simple aqueous bath. Cyclic voltammetric curves will be plotted to determine deposition potentials for nickel hydroxide. Effect of various preparative parameters such as deposition scan rate, deposition potential, concentration of the solution, bath temperature, pH of the bath, deposition time etc. will be optimized to get uniform and well adherent films. Previously, the advancement in science has taken place mainly with the discovery of novel materials. Characterization is an important step in the development of exotic materials. The X-ray diffraction (XRD) technique will be used for the phase identification. The surface morphology of the films will be studied using scanning electron microscopy (SEM). In order to study the interaction between electrode and electrolyte the surface wettability test will be carried out using contact angle meter. The electrochemical supercapacitor properties of the nickel hydroxide films will be studied by cyclic voltammetry (CV), charge-discharge and electrochemical impedance spectroscopy (EIS). During these experiments electrochemical cell consisting of platinum as a counter electrode and saturated calomel electrode (SCE) as a reference electrode in a suitable electrolyte will be used..

II. EXPERIMENTAL SET-UP

The experimental setup for the deposition of Nickel Hydroxide thin film is shown in figure 1. It exhibits the conventional three-electrode system with working electrode (cathode), counter electrode (anode) and reference electrode. The working electrode is well-clean stainless steel substrate of 1x5x0.05 cm^3. The counter electrode is polished graphite plate of dimension 2x2x0.5 cm^3. The saturated calomel electrode (SCE) is used as reference electrode. These three electrodes are fitted in the backlight holder having the slot for each electrode and the holder is fixed in cylindrical cell containing the electrolyte. The cylindrical cell is made up of glass for the purpose of visibility inside the bath and chemically inertness. All the depositions where carried out using automatic battery cycler (WBCS3000).

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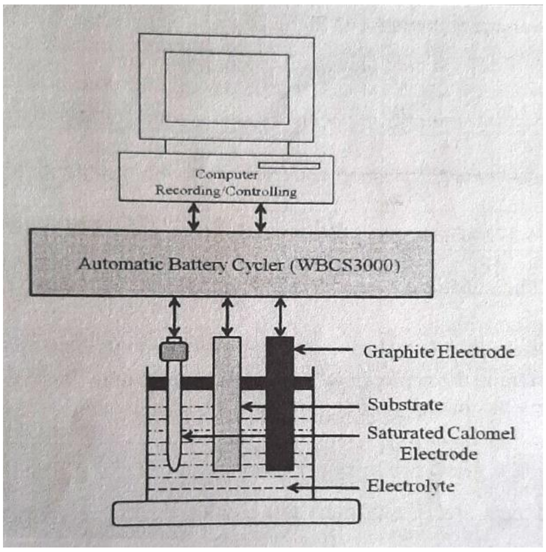

Figure 1: Schematic experimental setup for the electrodeposition of nickel hydroxide thin films

III. EXPERIMENTAL DETAILS

The deposition bath for Nickel Hydroxide thin film was made from an aqueous solution of 0.05 M Nickel chloride [$NiCl_2$] and 0.1 M Potassium Nitrate [KNO_3] maintained at room temperature (300 K). The Nickel Hydroxide film was deposited on commercially pure stainless steel (SS) substrate by potentiodynamic mode at scan rate of 100 mVs^{-1} in the potential range 0 to -1.2 V/SCE. The Optimized parameters for deposition of Nickel Hydroxide films are listed in table1.

Sr. No.	Film	Nickel Hydroxide
1	Medium	Aqueous
2	Bath Composition	0.05 M $NiCl_2$ + 0.1 M KNO_3
3	Potential Range (V/SCE)	0 to -1.2
4	Scan Rate (mVs^{-1})	100
5	Temperature (K)	300
6	Substrate	Stainless Steel (SS)

Table 1: Optimized preparative parameters for the deposition of Nickel Hydroxide thin films

IV. CHARACTERIZATION TECHNIQUES

The thickness of Nickel Hydroxide films was measured by weight difference method using sensitive microbalance. The structural analysis of the Nickel Hydroxide thin film was carried out using x-ray diffraction within the range 20-80^O on Philips PW-3710 using Cuk$_\alpha$ (λ=1.5409 A^o) radiations. The surface morphology films were analyzed by scanning electron microscope (SEM) (Model: JEOL JSM-6360) assembly. Contact angle measured by using Rame-hart model 500F1, USA.

V. CHARACTERIZATION TECHNIQUES

A. Cyclic Voltammetry Curves and Film Formation-

The cyclic voltammogram (CV) for SS substrate in 0.05 M $NiCl_2$ + KNO_3 electrolyte was carried out at room temperature. Electrodeposition from $NiCl_2$ was carried out potentiostatically following in parts the indications of Groza et al. Figure 2 shows the typical CV during deposition of Nickel hydroxide in the voltage range of 0 to -1.2 V/SEC at the scan rate of 100 mVs^{-1}. In this process nitrates ions from the bath, play a vital role in the deposition of Nickel hydroxide. Initially, when potential is applied to the deposition cell the reduction of nitrate ions take place and hydroxyl ions are formed. The standard reduction potential (E_0) of nitrate ion is-0.31 V/SCE.. The process during reduction is given as,

$$NO_3^- + H_2O + 2e^- \rightarrow NO_2^- + 2OH^-$$

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Furthermore, the sharp increase in the current was observed this may be due to the hydrogen evolution reaction which results into formation of molecular hydrogen and hydroxyl Groups.

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^-$$

These two processes are responsible for the enhancement of pH of the electrolyte close to the electrode. Hydroxyl groups formed via electrochemical reduction are expected to react with Ni^{2+} ions present in electrolyte. The cathodic peak is observed due to the reduction of Ni^{2+} species on the substrate. As a result, Nickel Hydroxide is deposited on the substrate according to the reaction given below-

$$Ni^{2+} + 2OH^- \rightarrow Ni(OH)_2$$

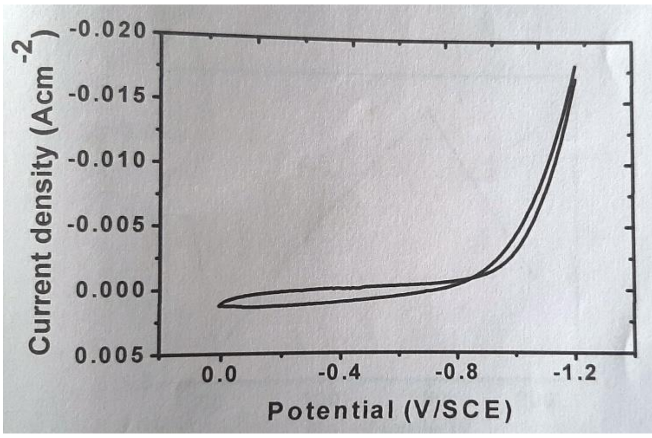

Figure 2: Cycle Voltammogram during nickel hydroxide film formation from aqueous bath at scan rate of 100 mVs^{-1}.

B. Thickness Measurements-

In this study of thin film materials, thickness of the film is most important parameter which alerts the properties of material due to surface phenomena. In the present study we have deposited Nickel Hydroxide thin films by varying number of potential cycles. The film thickness is measured in terms of weight deposited on unit area of substrate figure 3 shows the variation of mass deposited of Nickel Hydroxide with the number of deposition cycles as 50,100,150 and 200. From the figure3, it is seen that as the potential cycles are increased the deposited mass is also increased up to certain values and again it is decreased. This may be due to the development of tensile stress that tends to cause delimitation when film becomes thick. The film with maximum thickness 3.17 $mg.cm^{-2}$ has been used for further characterization and applications.

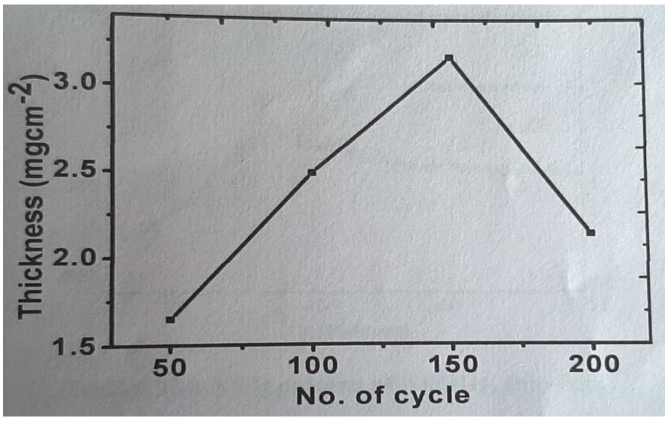

Figure 3: Variation of Nickel Hydroxide film thickness with number of potential cycles

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C. XRD Study-

For the structural clarification of the film, XRD was exploited in the 2θ range of $10-80^o$. Figure 4 shows the XRD pattern of the film onto the stainless steel substrate. The XRD pattern of the film revealed the formation of $Ni(OH)_2$ with hexagonal crystal structure [JCPDS card no. 14-0117]. Changyu Li et al shows the same crystal structure for $Ni(OH)_2$ material. The diffraction peaks at angles 2θ of 19.14, 33.88 and 37.83 are assigned to the (001), (100) and (101) planes of the $Ni(OH)_2$ crystal lattice, respectively. In addition with this, peaks originated due to stainless steel substrate material are indicated by asterisk '*'. Gund et al have reported similar kind of crystal structure for $Ni(OH)_2$ material prepared by the hydrothermal method.

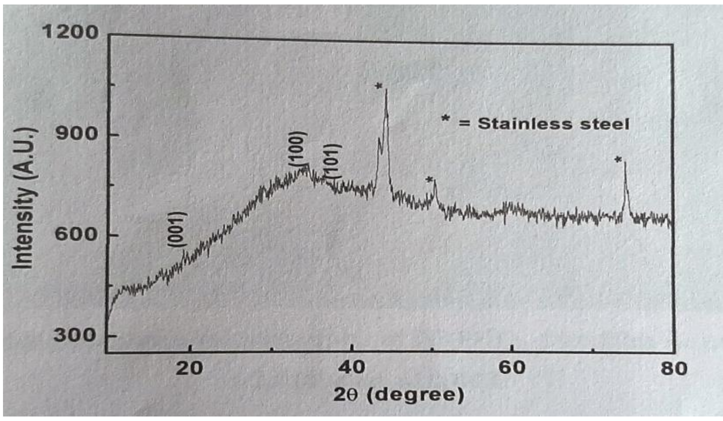

Figure 4: The XRD pattern of $Ni(OH)_2$ thin film

D. Surface Morphology Study-

Figure 5 shows the SEM images of $Ni(OH)_2$ thin film on the stainless steel substrate at magnification X2,000 and X10,000. The first are constituted of seemingly Compact Island that is separated by macroscopic cracks [figure 5(a)] in which one notices and interesting columnar growth as evidenced in the expanded. The Nickel Hydroxide film exhibited a highly porous sponge-like architecture [figure 5(b)], which provides plenty of interconnected channels for electrochemical active species to access the electrode surface and facilitates the ions transport. Such structure results in a large specific surface area and excellent electrochemical performance.

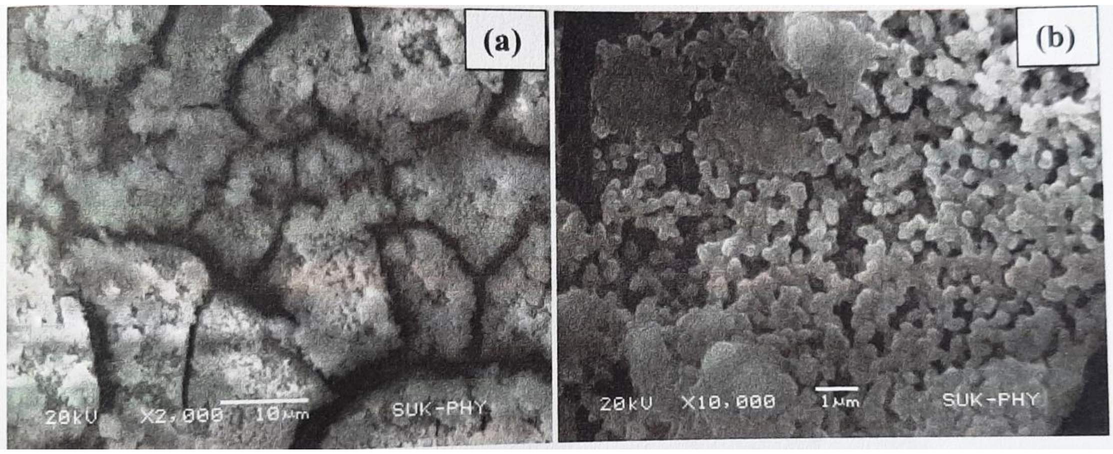

Figure 5: Scanning Electron Microscope (SEM) of $Ni(OH)_2$ thin film at magnifications X2,000 and X10,000

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E. Surface Wettability Test-

The wetting of solid with water, where air is surrounding medium, is dependent on the relation between interfacial tensions (water/air, water/solid and solid/air). The ratio between these tensions determines the contact angle (θ) between a water droplet on a given surface. A contact angle of 0^o means complete wetting and a contact angle of 180^0 corresponds to complete non-wetting. Both super-hydrophilic and super-hydrophobic surfaces are important for practical applications. Figure 6 shows the water contact angle measurement image of $Ni(OH)_2$ film. The observed water contact angle of $Ni(OH)_2$ was 65^o, it revealed the hydrophilic behavior of $Ni(OH)_2$ thin film (θ >90^o).

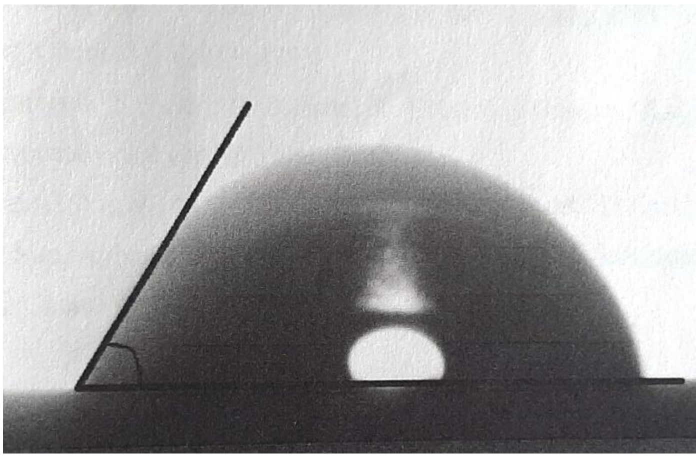

Figure 6: Measurement of Water Contact Angle of $Ni(OH)_2$ thin film

VI. CONCLUSION

This paper describes details of preparation of Nickel Hydroxide thin film using electrodeposition method. Also the various thin film characterization techniques like crystal structure determination, surface morphology, contact angle measurement etc, are discussed in this paper. The XRD patterns revealed the hexagonal structure of Nickel Hydroxide thin films. The SEM image shows the highly porous sponge-like morphology. The contact angle measurement shows the hydrophilic nature which is most important for the supercapacitor applications.

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