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# Morphological and Structural Properties of Cuo thin films Synthesised using Electrodeposition Method

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**ABSTRACT**

*Copper oxide thin films have been deposited on stainless steel substrates by using Electrodeposition. There has been a recent upsurge of interest in electrodeposition due to the use of metal deposition for the fabrication of integrated circuits, magnetic recording devices and multilayer structures. In this study, aqueous solutions of Copper acetate, sodium Hydroxide and Ammonia solution have been used as precursors during the deposition process. In this experiment, the Electrodepositions were done at a potential of −0.06V, −0.09V and−0.19V against the reference electrode for deposition times: 5s, 10s and 15s respectively. The morphological, compositional and structural characterization of the films were carried out by scanning electron microscopy, energy dispersive analysis and X-ray diffraction respectively. From SEM images, it shows that there were some variations on the surface microstructure depending on the deposition time and the grain sizes of the CuO nanostructures vary from a rough, inhomogeneous surface to a cloudy morphology with holes and cracks on the film's surface. The XRD patterns showed that the synthesized CuO nanostructures were crystalline. The crystallite size is varied between ~0.21- 0.46nm.*

**Key words:** Copper Oxide, Thin films, Electrodeposition, stainless steel

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## INTRODUCTION

The interest in the study of Copper oxide (CuO) is due to several reasons such as the natural abundance of starting material copper (Cu); the easiness of production by Cu oxidation; their non-toxic nature and the reasonably good electrical and optical properties exhibited by $Cu_2O$ [1].

Copper oxide can refer to: Copper(II) oxide (cupric oxide, CuO), a black powder or Copper(I) oxide (cuprous oxide, $Cu_2O$), a red or brown powder. Cupric oxide has a band gap of 1.1-1.9 eV and a monoclinic structure while cuprous oxide has a band gap of 2.1-2.6 eV and a cubic structure) [2-3].

Copper-oxide-based nanostructures have found applications in various areas including anode materials for lithium-ion batteries [4], antimicrobials [5] electrodes in dye-sensitized solar cells [6] heterogeneous catalysis [7] and magnetic storage media [8].

Different methods have been used to deposit CuO nanostructures such as, chemical bath method [9], sol–gel method [10] spray pyrolysis [11] reactive dc magnetron sputtering [12], SILAR, [13], thermal evaporation [14], among others.

The method adopted in this work is Electrodeposition. There has been a recent upsurge of interest in electrodeposition due to the use of metal deposition for the fabrication of integrated circuits, magnetic recording devices and multilayer structures. Electrodeposition or electroplating is the process of producing a coating, usually metallic, on a surface by the action of electric current. The part to be plated (*cathode*), and the desired metal which is to be plated onto the part (*anode*), are immersed in an electrolyte containing dissolved metal salts and other ions which allow proper flow of electricity through the solution. The 'redox' reactions lead to the deposition of the metal onto the part to be plated [15].

The electrodeposition process is well suited to make oxide films of metals such as copper, gold and nickel. The films can be made in any thickness from ~1µm to >100µm.

One of the advantages of electrodeposition is that it can form a material having a desirable form on a substrate. It also provides the deposit material with improved appearance and resistance to corrosion and abrasion to improve the thermoelectric characteristics of the material [16].

In this study, we report the preparation and deposition of CuO thin films on stainless steel slides substrates by Electrodepositin method and the effect of deposition time on the morphological and structural properties of the as-

prepared CuO thin films are investigated.

## MATERIALS AND METHODS

For the deposition of $Cu_X$ O thin film on stainless steel substrates, the substrate was first of all cut into smaller sizes and this was sandpapered and cleaned before the deposition. The stainless steel substrates were washed with detergent (soap solution), this was followed by cleaning the substrate in acetone and it was finally rinsed with distilled water. It was then hung in air for it to dry. The substrate was polished prior to the deposition process and for its surfaces not to be contacted by the solution was sealed using polyethylene terephthalete (PTFE) tape before the insertion into the vessel.

A conventional three-electrode system consisting of a work electrode, counter electrode, and reference electrode was used for the electrodeposition. The reference electrode was a saturated calomel electrode (SCE), the counter-electrode was made of carbon and the work electrode was a stainless steel substrate.

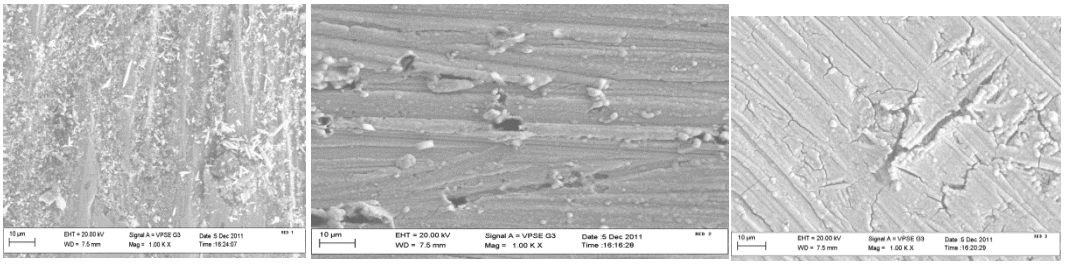
The solution for CuO deposition consists of 20ml of 0.1M Copper acetate, 5ml of 1M NaOH and 5ml of $NH_3$ solution. All reagents were of analytical grade and were used without further purification. All solutions in this study were prepared from distilled water and the electrolytic solution temperature was kept at room temperature during the experiments. The three electrodes were immersed into the electrolyte which contains a solution of the metal salt to be plated .The voltage of the D.C power supply and the current in the ammeter were 5V and 0.01A respectively. Electrodepositions were done at a potential of −0.06V, −0.09V and−0.19V against the reference electrode for deposition times: 5s, 10s and 15s respectively. After deposition, the films were rinsed with copious amounts of distilled water, dried with a hand drier.

## RESULTS AND DISCUSSION

### Morphological studies

An SEM analysis (x1, 000 magnification) of the final deposit shows that the morphology of the films obtained were rough, with grains, cracks and other imperfections.

The effect of the deposition time on the morphology of the films was observed. Fig. 1 (a-c) shows the scanning electron micrograph of films deposited at 5s, 10s and 15s respectively. It was seen from the SEM micrographs that film deposited at 5s shows a rough, inhomogeneous surface. As the deposition time was increased to 10s, the film showed a certain degree of cloudy morphology with holes on the film's surface. As the deposition time was further increased to 15s, it was observed that the film had a morphology which resembles "cracked-mud". Such "cracked-mud" morphology is used in supercapacitors.



**(a)** **(b)** **(c)**

**Fig. 1 (a-c) SEM micrograph of Electrodeposited CuO on stainless steel**

### Compositional studies

Fig 2 shows the EDAX spectrum of $Cu_xO$ deposited on stainless steel substrate using electrodeposition. The analysis shows the presence of copper (Cu), oxygen (O), and other elements like zinc (Zn) and iron which are from the stainless steel substrate.

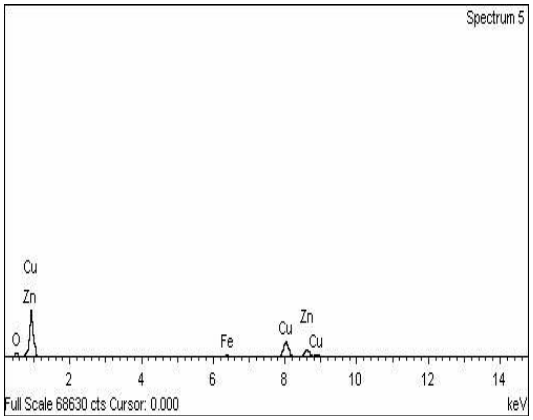


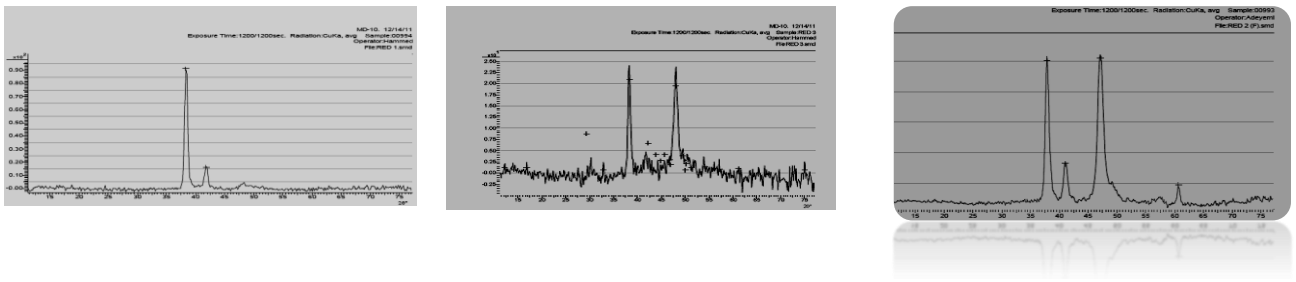

**Fig. 2 EDAX spectrum of $Cu_xO$ deposited on stainless steel using electrodeposition**

**Structural Studies**

In order to confirm the formation of $Cu_xO$, and to study the crystal structures of the resultant films, XRD analyses were employed. Fig. 3 (a-c) shows the XRD patterns of films deposited at 5s, 10s and 15s respectively.

The crystallite sizes were calculated by the X-ray line broadening method using the Scherrer formula: $D = k\lambda / \beta \cos\theta$, where λ is the wave length of radiation used (*CuK*α in this case), *k* is the Scherrer constant (0.94), β is the full width at half maximum (FWHM) intensity of the diffraction peak for which the particle size is to be calculated, θ is the diffraction angle of the concerned diffraction peak and D is the crystallite dimension (or particle size).

The effect of the deposition time on the structural properties of the films was investigated. At 5s, two sharp peaks were observed. These peaks corresponds to the (111) and (200) planes of $Cu_2O$. As the deposition time was increased to 10s, the number of peaks increased indicating higher crystallinity. As the deposition time was further increased to 15s the spectra showed that the synthesized oxide was copper chronium oxide ($CuCrO_2$).



(a) (b) (c)

**Fig. 3 (a-c) XRD pattern of the thin film deposited at 5s, 10, and 15s.respecetively**

From the above investigation, it is seen that the deposition time affects the crystallinity of the films. The crystallinity of the films increased as the deposition time increased. The average crystallite sizes of the films were 0.21nm, 0.373nm and 0.456nm for films deposited at 5s, 10s and 15s respectively.

## CONCLUSION

Electrodeposition method has been successfully used to synthesize and deposit CuO thin films on stainless steel substrates. These films were characterized using scanning electron microscopy (SEM), energy dispersive analysis (EDAX) and X- ray diffraction (XRD).

From SEM images, it shows that there were some variations on the surface microstructure depending on the deposition time and the grain sizes of the CuO nanostructures varies from a rough, inhomogeneous surface to a cloudy morphology with holes and cracks on the film's surface. The XRD patterns showed that the synthesized CuO nanostructures were crystalline. The crystallite size varied between ~0.21-0.46nm.

## REFERENCES


[1]. Papadimitropoulos, G., Vourdas N., Vamvakas V.E. & Davazoglou, D. (**2006**). Optical and Structural Properties of Copper Oxide thin films grown by Oxidation of Metal layers. *Thin Solid Films.* 515 (4):2428-2432.

[2]. Ray, S.C. (**2001**) Preparation of copper oxide thin film by the sol-gel-like dip technique and study of their structural and optical properties. Solar Energy Materials and Solar Cells, 68: 307-312.

[3]. Pierson, J.F., Thobor-Keck, A. & Billard, A. **(2003**). Cuprite, paramelaconite and tenorite films deposited by reactive magnetron sputtering, *Applied surface science* 210(3): 359-367.

[4]. Park, J. C.*;* Kim, J.*;* Kwon, H.*;* Song, H (**2009**). Gram-Scale Synthesis of $Cu_2O$ Nanocubes and Subsequent Oxidation to CuO Hollow Nanostructures for Lithium-Ion Battery Anode *Materials Adv. Mater.*, 21: 803–807.

[5]. Ren, G., Hu, D., Cheng, E.W.C. Vargas-Reus, M.A., Reip, P. Allaker, R.P. (**2009**). Characterisation of copper oxide nanoparticles for antimicrobial applications. *Int. J. Antimicrob. Agents*, 33: (6):587-590.

[6]. Liu, Y., Liao, L., Li, J. & Pan, C. (**2007**). Copper Nanocrystalline to CuO Nanoneedle Array: Synthesis, Growth Mechanism, and Properties. *J. Phys. Chem. C*, 111 (13): 5050-5056.

[7]. Li, G. H., Dimitrijevic, N. M., Chen, L., Rajh, T. &. Gray, K. A (**2008**). Role of Surface/Interfacial $Cu^{2+}$ Sites in the Photocatalytic Activity of Coupled CuO−$TiO_2$ Nanocomposites. *J. Phys. Chem. C*, 112: 19040–19044.

[8]. Kumar, R.V., Diamant Y. & Gedanken, A. (**2000**). Sonochemical Synthesis and Characterization of Nanometer-Size Transition Metal Oxides from Metal Acetates *Chemistry of Materials*, 12(8):2301-2305.

[9]. Dubal, D.P., Dhawale, D.S., Salunkhe, R.R., Jamdade, V.S & Lokhande, C.D. (**2010**). Fabrication of copper oxide multilayer nanosheets for supercapacitor application. *Journal of Alloys and Compounds* 492.

[10]. Wijesundera, R.P., Hidaka, M., Koga, K., Sakai, M., Siripala, W., Choi, J.Y., & Sung, N.E. (**2007**). Effects of annealing on the properties and structure of electrodeposited semiconducting Cu–O thin films. *Phys. Status Solidi B*, 244 (12): 4629-4642.

[11]. Parhizkar, M., Singh, S., Nayak, P. K., Kumar, N., Muthe, K. P., Gupta, S. K., Srinivasa, R.S., Talwar, S.S. & Major, S. S. (**2005**). Nanocrystalline CuO films prepared by pyrolysis of Cu-arachidate LB multilayers. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 257, 277-282.
[12]. Mugwang'a, F.K, Karimi, P.K. Njoroge, W.K. Omayio O. & Waita, S.M. (**2013**). Optical characterization of Copper Oxide thin films prepared by reactive dc magnetron sputtering for solar cell applications. *Int. J. Thin Film Sci. Tec.* 2(1):15-24.
[13]. Mitra, P. (**2010**). Preparation of Copper Oxide thin Films by SILAR and their Characterization. *Journal of Physical Sciences*, 14: 235-240.
[14]. Rao, G.N., Yao, Y.D. & Chen, J.W. (**2009**). Evolution of size, morphology, and magnetic properties of CuO nanoparticles by thermal annealing. *J. Appl. Phys.*, 105: 093901.
[15]. Schlesinger, M. and Paunovic, M. (**2000**). Modern Electroplating ($4^{th}$ edition), *Wiley, New York.*
[16]. Yang, J., Jin, Z., Chai, Y., Du, H., Liu T. & Wang, T. (**2009**). Electrodeposition of $CuInSe_2$ films by an alternating double-potentiostatic method using nearly neutral electrolytes. Thin Solid Films 517: 6617–6622.