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Synthesis and Characterization of some chalcogenide oxide Thin Films for Biosensor Application

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Abstract: This paper presents the synthesis and characterization of chalcogenide oxide (MgO, CuO, ZnO) thin film on tin-doped indium oxide (ITO) coated glass substrates via electrochemical deposition method. Here we use chalcogenide oxide as ZnO. The electrolytes used containing equimolar of zinc chloride (ZnCl2) and potassium chloride (KCl) ranging from 0.05 M to 0.2 M. The good structural properties of the ZnO thin film on the substrate was confirmed via high resolution X-ray diffraction (XRD). The fabricated thin films can be used for many applications like chemical sensor, biosensor, solar energy, etc. but in my project, I will use it for biosensor application.

Keywords: Electrochemical Deposition, Zinc Chloride, XRD, Potassium Chloride, Biosensor etc.

I. INTRODUCTION

A biosensor is an analytical device, which converts the modification of the physical or chemical properties of a bio-matrix (e.g., enzyme, antibodies, receptors, organelles, microorganisms) into electric or other kinds of signal whose amplitude depends on the concentration of defined analysts in the solution [1]. They are becoming essential in the field of healthcare, chemical and biological analysis, environmental monitoring and food processing industries. According to the receptor type, biosensors can be classified as enzymatic biosensors, Genosensors, Immuno sensors etc. [2]. Biosensors can be also divided into several categories based on the transduction process, such as electrochemical, optical, piezoelectric, and thermal or calorimetric biosensors[3]. ZnO is wide band gas semiconducting oxide material which is biocompatible with a high isoelectric point of 9.5 (suitable for adsorption of protein shaving low isoelectric point (IEP)(and has specific properties including ease of fabrication, high specific surface area, nontoxicity, chemical stability, strong adsorption ability and high electron communication feature which make itan excellent matrix to be used in biosensors. ZnO thin films and nanostructures have been extensively used for the detection of various biomolecules [4-9]. ZnO with various nanostructures prepared by different fabrication techniques has been widely used for enzyme immobilization in recent years. Recent advances in biocompatible nanomaterial and biotechnology open a promising field toward the development of biosensors [5].

II. ELECTROCHEMICAL DEPOSITION OF 1-DIMENSIONAL ZNO NANOMATERIAL.

The electrochemical deposition can be explained by understanding mechanism of potentiostat or galvanostat (able to generate a constant current between electrodes) or coulostat(able to maintain a constant charge). In this study, only potentiostat will be discussed. In addition, electrochemical deposition can be described as the flow of current among electrodes and conducting material including ionically conducting phase called an electrolyte solution. Unlike electrochemical deposition without external current flow, a current flow by potentiostat or galvanostat results in significant changes of grown crystals. There are three electrodes connected to the potentiostat for fabricating ZnO nanomaterial. They are working, counter, and reference electrodes [5-6]. The materials for reference, working, and counter electrodes used in this study are silver/silver chloride (Ag/AgCl), ITO coated glass (or conducting substrates such an ITO coated plastic substrates), and platinum (Pt), respectively[7-8]. These electrodes are critical for formation of nanomaterial in aqueous solution at a constant electric field, especially depending of the reference electrode due to its own potential in the ionically conducting phase. By applying electric field between electrodes, charged particles (ions) are accelerated towards one of the two electrodes and establish a field with the direction depending on the sign of the charged particle. Only resistance for the charged particles can be caused by the viscosity of the medium where the particles are moving in this study, the conducting medium (phase) is a water-based solution which viscosity is 1 with zinc (Zn+) and potassium (K+) and chlorine (Cl-) ions. An electrical voltage is applied to these electrodes and the ions start movement. Even though some of the colored ions can be recognized at the one of electrodes, the resulting crystal

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at a working electrode is white coloredZnOnanomaterials with various lengths (50 nm) depending on growth time[9]. The reaction occurred at the each electrodes can be described as

$$\text{Reference(Ag/AgCl) electrode: } Ag + Cl^{-} \longrightarrow AgCl + e \quad \ldots\ldots\ldots\ldots\ldots\ldots(1)$$

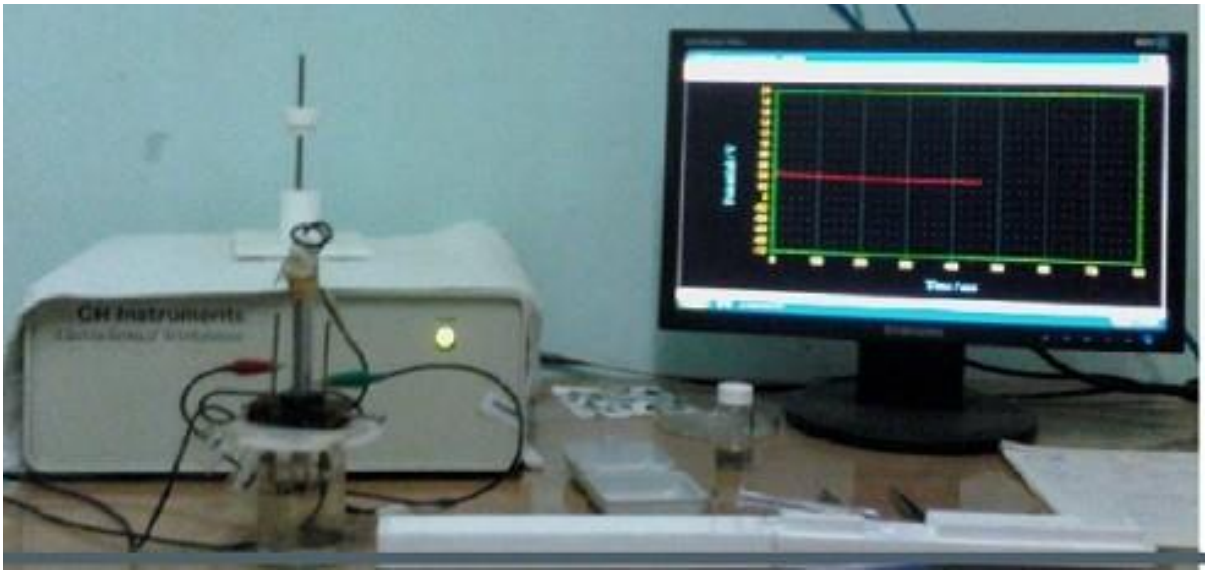
$$\text{Counter(Pt) electrode: } 0.5O_2 + H_2O + 2E \longrightarrow 2OH \quad \ldots\ldots\ldots\ldots\ldots\ldots(2)$$

$$\text{Working(ITO coated glass)electrode } Zn + 2(OH^{-}) \longrightarrow ZnO + H_2O \quad \ldots\ldots\ldots\ldots\ldots\ldots(3)$$

At the counter electrode, additional oxygen is achieved by bubbling gas directly into the aqueous solution prior to reaction continues to end of the reaction. The application of an electric field, reduction of oxygen occurs, leading to the production of OH- ions on the surface of the working electrode as well as in the solution.

III. ELECTROCHEMICAL SYNTHESIS SET-UP

The electrochemical synthesis setup is as shown in Fig 1. It consists of CHI 660: A Computer controlled electrochemical polymerization system (Potentiostat/ Galvanostat), and three electrode glass cell. The three electrode glass cell consists of working electrode ITO coated glass Pt as counter electrode and AgCl as reference electrode and electrolyte solution.



.Fig. 1CHI 660 Instrument Electrochemical Work Station used for the

Synthesis of ZnO thin films

IV. MATERIAL AND METHODS

ZnO nanostructures were grown by electrochemical deposition methods in electrolyte containing $ZnCl_2$ and KCl ranging from 0.05 M to 0.2 M with increment of 0.05 M per experiment. The molar ratio of $ZnCl_2$ and KCl was kept at 1 throughout the experiment. The electrode position of ZnO nanostructures were performed on the conductive side of the ITO coated glass slides, which acted as the working electrode while copper wire as counter electrode. Prior to Electrode position, the ITO substrates were cleaned in acetone, methanol and de-ionized (DI) water for 5 minutes each in an ultrasonic bath and dried under air flux. The copper wire was sanded by abrasive paper to remove the native oxide layer and then rinsed with isopropanol. A beaker filled with electrolyte was immersed in a water bath where the whole setup was heated on a hotplate at 90. Then the Electrode position was carried out with voltage of 8V using a Keithley 2400 source measure unit (SMU) that interfaced to a computer[10-12]. XRD patterns were used to determine the structural properties of the samples.

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V. WORKING OF BIOSENSOR

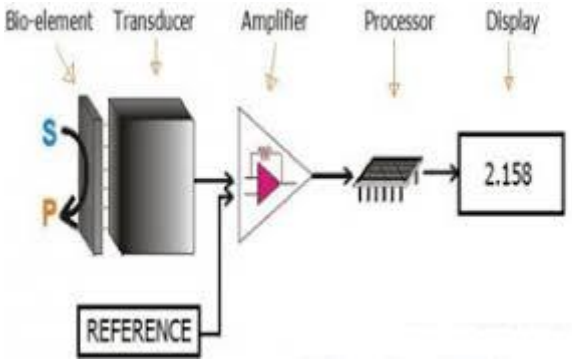


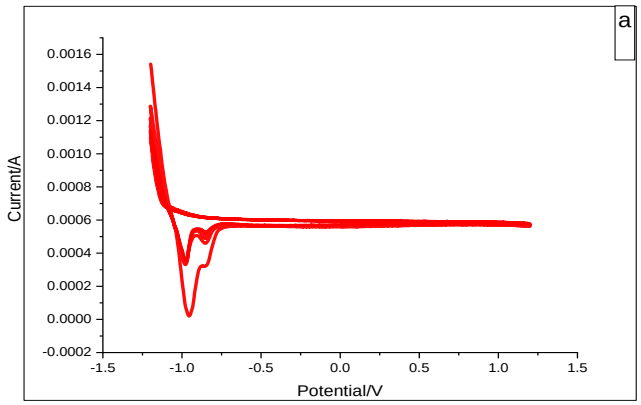
Figure 2: Schematic Diagram of biosensor

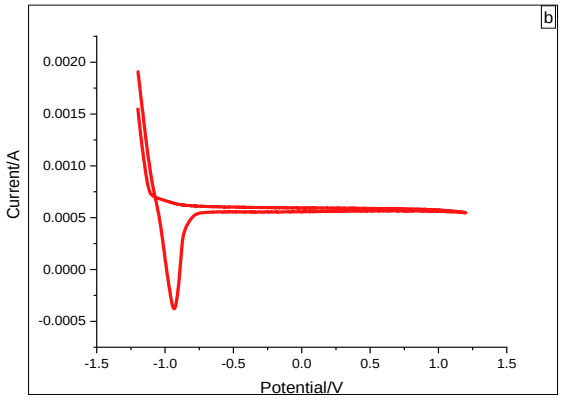
Biosensor is an analytic device that measures concentration of an analyte. In Biosensor, a biological material (such as enzyme, antibody, whole cell, nucleic acid-RNA, DNA etc) is used to interact with the analyte. This interaction produces a physical or chemical change, which is detected by the transducer. This change is converted into biological or chemical reactions by generating signal proportional to the concentration of an analytic in the reaction. This change is converted into electrical signal which is amplified by an amplifier. This amplified electrical signal is then processed by processor and converted in to analyte concentration present in the solution. This is displayed on the screen of the Biosensor[13-15].

VI. CYCLIC VOLTAMMETRY (CV) STUDY

Cyclic Voltammetry (CV) measurements were done to examine the electro catalytic activity of prepared ZnO. Fig. 3 shows the Cyclic Voltammetry (CV) plots of prepared ZnO electrode at the different deposition time 80 min, 100 min, 120 min respectively at a scan rate 0.05 V/s. The electrolyte used for these measurements is ZnCl2 and KCl solution with 0.1 M concentration. The glucose concentration in electrolyte is varied Ranging from 0.05M – 0.2M at Initial [16-17].

E(V) = −1.2, High E(V) = 1.2, Low E(V) = −1.2, Scan Rate (V /s) = 0.05.





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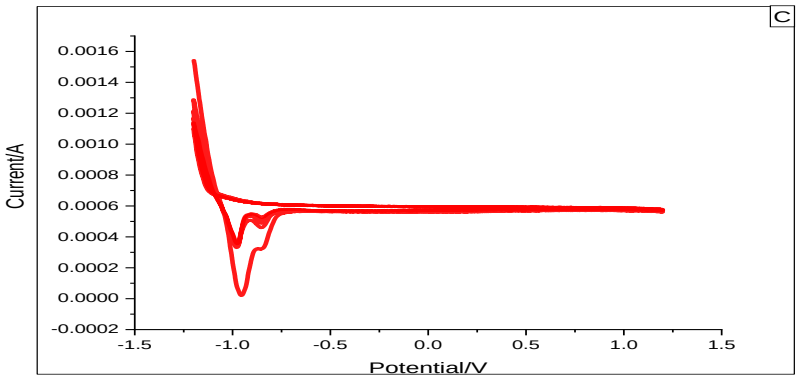

Figure 3: Cyclic Voltammograms of Electrochemical Deposition Methods of ZnO Thin Films in an Electrolyte Containing ZnCl2 and KCl Ranging from 0.05M – 0.2M at Initial E(V) = −1.2, High E(V) = 1.2, Low E(V) = −1.2, Scan Rate (V /s) = 0.05.

VII. X-RAY DIFFRACTIONANALYSIS

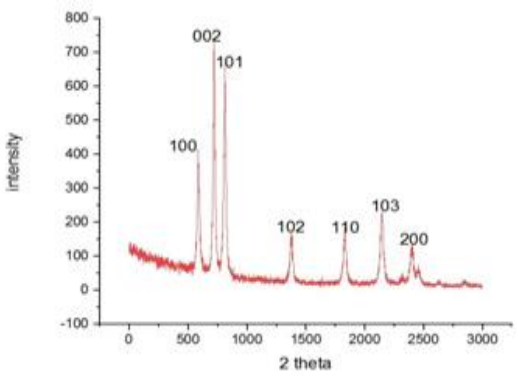

Figure 4: XRD pattern of ZnO Thin film

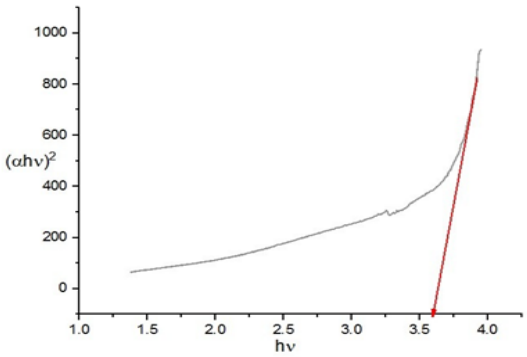

Figure 5: Plot of (αhν)2vs(hν) Curve of ZnO Thin Film

The XRD pattern of ZnO thin film prepared by electrochemical method on glass substrates is shown in Figure (4). All the peaks of the ZnO thin films matches with correspond to the peaks of standard ZnO (JCPDS 36- 1451) For the samples, different diffraction peaks are observed in the XRD pattern, showing the growth of ZnO crystallites along different directions. Strong preferential growth is observed along (002) plane indictating that the films are oriented along c-axis [18]. The typical hexagonal wurtzite structure of thin films is inferred from the XRD pattern. The crystallites sizes (D) of the films are estimated using the Scherer formula

$$D = \frac{0.9 * \lambda}{\beta_{2\theta} \cos\theta} (4)$$

where λ is the wavelength of X-Ray used λ = 1.54A and $\beta_{2\theta}$is the full width at half maximum of(002) peak of XRD pattern Bragg angle, $\beta_{2\theta}$is around 36.4. The average value of grain size is found to be 28.92nm. The evaluated structural parameters of thin films are calculated. The lattice parameters are found as the following (a= b= 3.257(A^0)) and (c = 5.25A^0)

(a = b)(A^0)		c (A^0)	
Calculated	Standard	Calculated	Standard
3.255	3.253	5.217	5.215

Table 1. Lattice parameters of the ZnO thin film

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The optical band energy gap E_g of the thin film was calculated from plot as shown in figure 5 above. The presences of single slope in the plot suggest that the film have direct and allowed transition. For such transition we have $(\alpha hv)^2 = A(hv - E_g)$ where (α) is absorption coefficient, (hv) is the incident photon energy, (E_g) represents the optical band gap and A is constant. The band gap energy is obtained by extrapolating the linear portions of the plot to zero absorption coefficient. The band gap value of *ZnO*thin film is found to be 3.6 *eV* . These values are in good agreement with the values reported by others[19-20].

VIII. CONCLUSION

We have successfully synthesized ZnO thin film using Electrochemically method on ITO coated glass substrates. It is observed that the synthesize sample having Hexagonal wurtzite structure of nanocrystalline thin films found crystallite size 28.92nm and energy band gap 3.6 eV. Electrolyte containing of $ZnCl_2$ and KCl and have been synthesized which sending glucose biosensor for detection glucose in human blood sample

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