

# Behavior of Ni in ZnO/Al working electrode for K<sup>+</sup> ion sensor

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**ABSTRACT**---Ni doped ZnO has been widely studied as a diluted magnetic semiconductor (DMS), but in this work we have shown its use as a biosensor. To analyze biological samples, it is required to convert the biological events to an equivalent electrical output, which is done by electrochemical biosensors at an affordable price. Alkali metal ions play a vital role in biological events and study of which is necessary to know the healthy functioning of human body organs. Potassium ion (K<sup>+</sup>) is one of the alkali metal ion in blood which needs regular monitoring. This was a motivating factor for us to go for the fabrication of working electrodes like ZnO coated on Al substrate (ZnO/Al) and Ni doped ZnO coated on Al substrate (Ni in ZnO/Al) in sol-gel method by Chemically Wet and Dry technique (CWD), an indigenous method that has been developed in our lab. The fabrication process was followed by in-situ annealing of the sample electrodes at 450°C in presence of Nitrogen environment. The crystalline property of the synthesized materials was confirmed by XRD study. The Hall Effect study shows higher mobility and conductivity values for Ni doped ZnO in comparison to pure ZnO. The pH level variations for various concentration of KOH solution were studied by both electrodes separately considering Ag as reference electrode. The sensitivity of Ni in ZnO/Al working electrode was found to be more stable and sustains for a longer period than that of ZnO/Al working electrode in terms of potassium sensing.

**Keywords**-- Working electrode, Sol-gel chemical wet and dry (CWD), electrochemical K<sup>+</sup> sensor

## I. INTRODUCTION

One of the most notable minerals in our body is Potassium (K<sup>+</sup>), as it keeps our heart as well as kidney function normal. Not only potassium but also sodium helps in regulation of water and acid-base balance in blood and different tissues in our body. At the same time potassium has effectively initiates sodium-potassium exchange on cell membranes which is responsible for electrical signal that carry out the nerve impulses.

Since sodium and potassium are present in their ionic form in our body, hence the electrical conductivity mechanism in the body involves ions as charge carrier. Therefore, by transducing ionic currents picked up due to physiological activity, electric currents now can be analyzed by electronic instruments [1]. This electrolyte ion conducts electricity in the biological systems along with other alkali metal ions. So, the K<sup>+</sup> ion is most useful for physiological functions of nerves and muscles throughout the body. The root cause of several diseases can also be due to the abnormality of potassium ion concentration in blood. So, monitoring the K<sup>+</sup> concentration has become

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an important challenge. There has been extensive research about potassium ion sensors based on ion-selective electrodes [2]. For decades now, it has been the goal of various researchers to manufacture an electrode that would be quite sensitive, can be reproduced and is able to withstand repeated experiments for a longer period. Nowadays, researchers prefer ZnO material for chemical sensing purpose and using this material biosensors can be fabricated. ZnO has some remarkable properties like harmless in nature, bio-safety, agreeable to bio-molecules, upgraded analytical performance, highly sensitive, high electron transfer rate, easily fabricated, low cost and used to detect analytic components such as cells, protein, nucleic acids [3]. Zinc oxide [ZnO] is an intrinsic n-type belonging to II-VI group of semiconductors with wide bandgap energy (3.37 eV) and large excitation binding energy (60 meV) at room temperature [RT] [4-10]. Thus, it is considered as promising candidate for optical and optoelectronic applications in nanoscale devices [8,9]. It is used as a transparent electrode in solar cells [10], chemical and gas sensors [11,12], and spintronic devices [13]. Again, ZnO has high ionic bonding about 60% and at biological pH values it dissolves very slowly. These characteristics enable ZnO to produce stable signals with reproducibility when used in biosensor [14]. Pure ZnO is a good insulator; however, by adding specific impurities and by heat treatment its electrical conductivity can be increased. Earlier the working principle of sensing electrodes in an electrochemical cell involved the development of an electric potential when the opposite sides of the thin glass membrane come into contact with two liquids of different ionic concentrations [15, 16]. Here, we have reported a non-glass ZnO film coated on Al electrode whose potential is measured with Ag as the reference electrode. In this study we have discovered that the resistivity of the ZnO film decreases when coated on metallic electrode like Al. The working electrode (or sensing electrode) produces the electromotive force because of ion exchange in the electrolyte solution. Hence by measuring the electromotive force, we can estimate the ionic concentration of the solution. By taking potassium hydroxide solution (KOH) of different concentrations the efficiency of electrodes (both undoped and doped ZnO on Al substrate that are fabricated in our lab), can be studied to sense potassium ion. The detailed discussion about  $K^+$  sensing by taking ZnO as coating material on Al and Ag electrodes has been reported by us earlier [17].

Here in this paper, section 2 briefly discusses about the theoretical concept of metal-semiconductor-electrolyte interface, section 3 involves the experimental procedure and setup, section 4 contains the results and discussion and section 5 concludes the paper.

## II. THEORY

To extract and analyze biological signals with the help of electronic instruments, it's necessary to transduce ionic currents into electric currents. For that purpose, electrodes are required to be in contact with aqueous ionic solutions of the body [1]. One of the most important component of a biosensor is the working/sensing electrode. In an electrochemical system, working electrode is the electrode on which the reaction of interest takes place. One must be careful while selecting the appropriate electrolyte as well as the working (sensing) electrode. By doing so, one can target a specific bio molecule.

If a semiconductor coated metal is being used instead of pure metal as working electrode, then it is quite simple to vary its electrochemical response extensively, by changing the impurity levels of the semiconductor material. This is one of the greatest advantages of semiconductor over metal as a sensing electrode. This property of

semiconductor electrodes has potential applications in developing bio-sensors [18]. Furthermore, it is a difficult task to make ohmic contacts on the semiconducting material for electrical signal detection. We therefore go for semiconductor coated metal instead of semiconductor only electrode. In order to use a semiconductor coated metal electrode in an electrochemical cell, the two most important parameters to know about are the Fermi level or  $E_F$ , (which is defined as the energy level where the probability of an electron occupying is one half), and the corresponding electrochemical potential ( $\mu_e$ , redox) of electrons in solution. The value of this potential is given by the Nernst equation

$$E_{redox} = E_{redox}^{\circ} + \frac{RT}{nF} \ln \left[ \frac{C_{ox}}{C_{red}} \right] \quad \text{Eqn. 1}$$

where,  $C_{ox}$ = concentrations of oxidized species

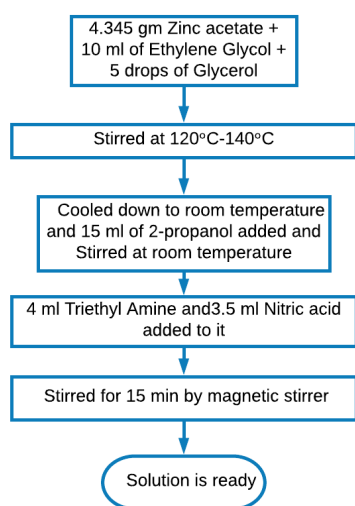
and  $C_{red}$ =concentrations of reduced species

Now the electron energy levels in the solid can be related to liquid phases. In solid-state physics, the reference is electron energy in vacuum, while in electrochemistry the reference is the standard hydrogen electrode (SHE). It has mostly been proved that SHE appears to lie at -4.5eV with respect to vacuum level. From the theory of electrochemical half-cell it is known that when a semiconducting electrode dipped in a solution (electrolyte) a difference in potential takes place at the interface of the two phases. This difference in potential can be measured by using Nernst equation. The metal-semiconductor-electrolyte Schematic band diagram has been discussed in detail in literature [17]. Here a drop in potential takes place at both the boundary layers. The boundary layers include both the interfaces. One of the interfaces is between metal and semiconductor and the other one is present between semiconductor and the electrolyte. Due to this difference in potential, when a semiconductor coated metal is dipped in the redox electrolyte (KOH) there is the flow of charge from one phase to another phase takes place to maintain equilibrium. That results in band bending. In this case charge exchange takes place between the conduction band and valence band electrons of the semiconductor and the electrolyte ions as well, due to chemical reaction. Then an electrostatic field has been developed due to charge separation among the charged particles. This field creates a potential difference as found in literature [19]. At the semiconductor-metal interface there is exchange of electrons between conduction and valence bands in the semiconductor and metal takes place as it is an ohmic interface.

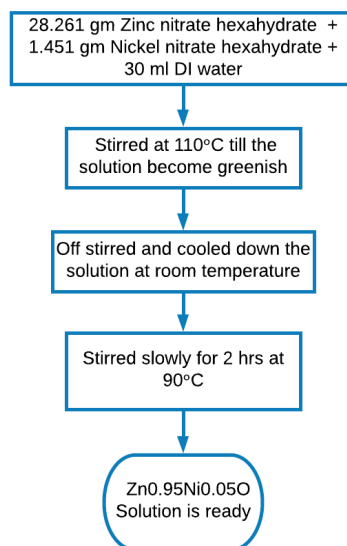
### III. EXPERIMENTAL METHOD AND SET UP

#### 3.1. Preparation

The sol-gel process was used to prepare pure ZnO and Ni doped ZnO ( $\text{Zn}_{1-x}\text{Ni}_x\text{O}$ ,  $x=5$  mol %) solutions and from these solutions thin films on Al substrates were deposited by dip coating CWD (chemical wet and dry) method followed by *in situ* annealing. Taking Zinc acetate as precursor, from ethylene glycol and glycerol, ZnO solution was prepared in the lab by sol gel method as shown in Fig.1.



**Figure 1:** Flow chart for preparation of ZnO solution by sol-gel method

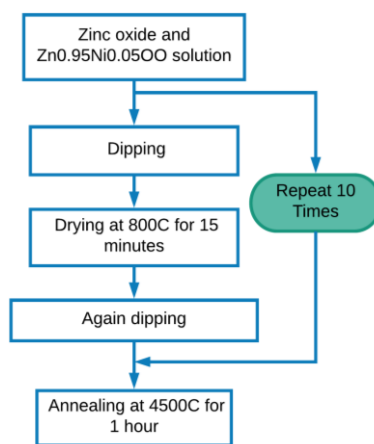


**Figure 2:** Flow chart for preparation of Zn<sub>0.95</sub>Ni<sub>0.05</sub>O solution by sol-gel method

In the similar manner Ni doped ZnO solution was prepared from precursors Nickel nitrate hexahydrate [Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O] and Zinc nitrate hexahydrate [Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O] by sol-gel method which is shown in Fig. 2.

### ***Film Deposition***

The desired thin films were coated on Al substrates from the ZnO and Zn<sub>0.95</sub>Ni<sub>0.05</sub>O solution by chemical wet and dry (CWD) dip coating method. Al sheets of dimension (7.5 cm × 2.5 cm × 1 mm) were used as substrates. First the substrates were polished and cleaned with acetone. Then, dipped in a beaker containing diluted nitric acid (5:1 proportion) for a fraction of second followed by ultrasonic cleaning in 2-propanol at about 30<sup>0</sup>-40<sup>0</sup>C for 15 minutes. The cleaned substrates preheated at 40-50<sup>0</sup>C for 2 minutes then clamped in the sample holder. The beaker containing the solution to be coated kept under it. Then with the help of a CWD set up the Al substrates were coated. The coatings were dried at 80<sup>0</sup>C and then burnout at 200<sup>0</sup>C in situ for half an hour. After that annealing at 450<sup>0</sup>C for 1 hour was done. Figure 3 shows the flow chart for ZnO and Zn<sub>0.95</sub>Ni<sub>0.05</sub>O coating on Al substrates by using CWD technique. The detail description of chemical wet and dry (CWD) technique has been described in our earlier reported work [20,21].



**Figure 3:** Flow chart for ZnO and Ni doped ZnO thin film

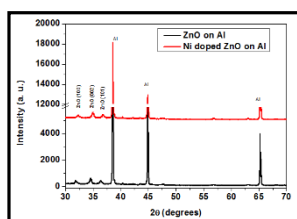
### *Experimental detail*

The experimental set up consists of two electrodes, one is sensing or working electrode and other is reference electrode. The experimental setup has resemblance with half-cell electrochemical model having two electrodes as explained in one of our literature [17].

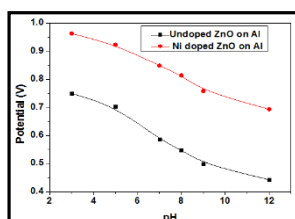
The electrodes were immersed in the electrolyte (KOH) solution which was kept in the beaker. The sensing electrodes (or working electrode) were ZnO/Al and Ni in ZnO/Al. The reference electrode was chosen to be a pure silver wire. The potential and the respective current of the electrochemical cell were extracted using data acquisition system NI PXI-1042 with data acquisition card no. NI PXI-4072.

## **IV. RESULTS AND DISCUSSION**

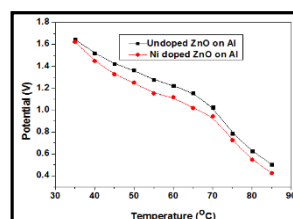
Fig. 4. Shows the X-Ray diffraction (XRD) patterns of both the ZnO and Ni doped ZnO films on Al substrates. The patterns clearly indicate that both the samples are polycrystalline nature. Both the XRD patterns show peaks of ZnO as well as Al. There are no other impurity peaks found excluding Ni in the XRD pattern. This indicates Ni atoms are completely incorporated in host ZnO sample. The Wurtzite structure of ZnO is confirmed from its major peaks. These peaks are matched well to the JCPDF file of number 890510. By using WinCell XRD software the lattice parameters 'a' and 'c' of ZnO film coated on Al wire were estimated to be 3.3638 Å and 5.3845 Å respectively which were slightly higher than the lattice parameters of ZnO i.e., (a = 3.251 Å and c = 5.234 Å). Similarly the lattice parameters for Ni doped ZnO on Al substrate are found to be a = 3.193 Å and c = 5.1138 Å respectively. However, the c/a ratio of both are (1.6037 and 1.6015) is in consistent with the reported value of pure ZnO (1.6099). Furthermore, we have calculated the volume of Ni doped ZnO (156.40 Å<sup>3</sup>) which is smaller than volume of undoped ZnO (164.23 Å<sup>3</sup>). This variation in volume occurs as the Al atoms replace the Zn atoms at the interface region. In addition to this, the effect of the Al metal taken as substrate has been reported by us earlier [22].



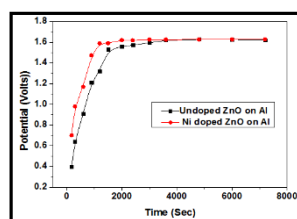
**Figure 4:** The X-ray diffraction pattern for ZnO and Ni doped ZnO thin film on Al substrate



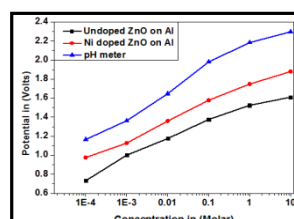
**Figure 5:** Comparison graph of ZnO film and Ni doped ZnO coated on Al electrodes for variation of pH values.



**Figure 6:** Effect of Temperature on the EMF response to K<sup>+</sup> standard solution containing KOH.



**Figure 7:** The response Time of both undoped and Ni doped ZnO coated Al electrodes



**Figure 8:** Comparison graph of Ni doped ZnO film and undoped ZnO film on Al electrode for K<sup>+</sup> concentrations ranging from 1μM to 10M

### 6.1. Effect of pH

The performance of pH sensors depend on the electrochemical potential of the electrodes. The density of surface sites on which the pH dependent surface potential is formed add significant constraint for the ion-sensitive layers. Both ZnO/Al electrode and Ni in ZnO/Al electrodes can be used as pH sensor and their application is based on the reaction takes that place at the metal-semiconductor-electrolyte interface. Here we have taken different pH solutions with pH ranging from 2 to 12 for testing the effect of pH on the output response of both the working electrodes. As can be seen from Fig.5, the response to pH has a decreasing trend as we go from acidic towards base region. A slightly steeper decrease is noticed between pH 6 to 8 for both the electrodes. It shows that both the sensors are slightly more sensitive in the base region. This property is very useful for biosensors. However in comparison to ZnO/Al electrode, the Ni in ZnO/Al working electrode is showing better result. This proves its usefulness for biosensor applications.

### 6.2. Effect of Temperature

We have varied the temperature from 30°C to 90°C to check the effect on the potential of potassium ion sensors, the result of which is shown in Fig. 6. With increase of surrounding temperature, the electrochemical potential response of both electrodes decreases. From the results it is evident that response of both the electrodes are better at room temperature in comparison to high temperature.

### 6.3. Effect of Timing

The time response of the proposed ZnO/Al electrode and Ni in ZnO/Al electrode were studied from this experiment whose graph is shown in Fig. 7. The electrolyte here we had used was Potassium hydroxide solution (KOH) of 1M concentration and the electrodes were inserted into it. The potential was measured with NI PXI-1042 DAS with the NI PXI-4072 data card. The data were taken with continuous supervision for 20 days. The gradual increase of potential was noticed which took half an hour to get stabilize. It is also found that in comparison to ZnO/Al electrode, Ni in ZnO/Al electrode shows better time response.

### 6.4. Effect of concentration

To accomplish our requirement i.e. to evaluate the ion-sensing ability of both the working electrodes i.e., ZnO/Al electrode and Ni in ZnO/Al electrodes were immersed in KOH solution, which was taken as electrolyte one by one. The molarity of KOH solution was varied from 1  $\mu$ M to 10M. Here the KOH solution is preferred as electrolyte because of the larger radius (0.138 nm) of the  $K^+$  ion than the  $Zn^{2+}$  (0.074 nm). Thus the possibility of its incorporation into the host lattice is less. The charge concentration around the working electrode gives the output voltage. Fig.8 shows response of above-mentioned working electrodes to sense the  $K^+$  ion concentration from the electrolyte and for comparison purpose the pH meter reading has been plotted. However, almost linear response is shown by these electrodes in this range. As can be seen from the graph, the sensitivity of Ni in ZnO/Al is more than that ZnO/Al electrode.

## V. CONCLUSION

In this paper, ZnO/Al electrode and Ni in ZnO/Al electrode have been fabricated by CWD technique. These prepared samples annealed at 450°C for 1 hour and then used as working electrodes. From XRD analysis Wurtzite structure of ZnO was confirmed. Various experiments were conducted with respect to pH level, temperature, time and concentration to evaluate the performance of both the electrodes. Their results showed the capability of the electrodes for sensing  $K^+$  ion. However the doped electrode shows better characteristics than undoped one. Thus, these electrodes can be used as biosensor to sense  $K^+$  ion in biological field.

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