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ZNO Gas Sensor Thin Film Preapared by Sol-Gel Dip Coating Method

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Abstract:- *The preparation of Zinc Oxide thin film with dip coating of gas sensing methods are we discuss in this paper .For sol gel preparation the researcher using Zinc Nitrate hexahydrate (Zn $(NO_3)_2$ .6 H_2o) and organic polymer sodium carboxy methyl cellulose (Na-CMC) as basic material from the size of the prepared thin film varied by XRD, morphology was studied by (FE-SEM) and the gas sensing was studied using acetaldehyde has been tested. The gas sensor parameter such as response selectivity, response/recovery time towards acetone were reported.*

Keywords: *ZNO, Na-CMC, Sol- gel dip coating, thin film, acetone alcohol sensors.*

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I. Introduction Section

The thin film nano material surface volume will more effects (C.S. Prajapati & others 2013)[*2].The nano material have unique separate physical, chemical and optical properties (B.Lyson-sypien & others,2012)[*3]. Now a days we use nano material in the field of gas sensors it help in toxic and combustible gases (P. Rai & others, 2013)[*4].The excess of toxic and combustible gases cause humiliation to Environment and are hazardous to human health. Hence fabrication of gas sensor with good sensitivity, selectivity, quick response and recovery time to the lowest concentration of target gas together with low operating temperature is an art of interest. Semiconducting metal oxide nano particles are extensively studied for gas sensing application due to small dimension, suitable operating temperature, more surface sites available for gas adsorption, high response towards many gases, low cost, portability, non-toxicity etc., [R.C. Pawar & others ,2013][*5,6].

A wide band gap of Zinc Oxide intrinsic n-type semi conductor. It is one of the broad studied material for sensing application (S. Ozturk & others ,2011)[*7].It have given best advantage s like nano-toxic nature electron mobility abundant availability and high chemical stability (P.P. Sahay,& others ,2008)[*8-10]. It can be prepared by single crystal also powder thin film (M. Aslam & others ,2014)[*11-13],IT also have good high sensing response from oxidizing reducing gases (N. Hongsith & others ,2008)[14].The film extremely depend on the surface state and the morphology of semiconductor metal oxide(Y. Lv & others ,2009)[*15]. It act as a chemical reaction center (C.M. Chang & others ,2010)[*16-18]. In order to modified the surface state to preferable for sensing application from sol-gel dip coating so the researcher could chosen thin film deposition technique and works.

The Reason that the researcher could chose dip-coating is a simple, cost effective and also the wed chemical method which widely used for Zno (T.T. Trinh & others ,2011)[*19]. From this method the uniformity, thickness, porosity and morphology are altered by controlling sol-gel concentration, withdraw speed timing and dipping and drying time (K. Thongsuriwong & others ,2013)[*20]. In our present work dip coating was prepared by automated dip coating units. After sol prepared ,thin film coated followed by heating furnace are step followed in sol-gel dip coating method (C.H. Chia & others ,2013)[*22].The glueness of the coated film on the substrate depend upon viscosity of sol. The Na-CMC is used for sol-gel preparation because of it film forming ability due to rich OH –group (K. Shao & others ,2008)[*23] and its thickness property are also characteristic. The Na-CMC is an in organic green polymer. Which can be soluble in di-water (J. Chen & others,2008)[*24]. Emulsifier, builder stabilizer are general utilities of Na-CMC (H. Kono & others, 2014)[*25-26].

The most main properties are depend on molecular weight and degree of substitution for (example): No of OH- group substituted per an hydro glucose units (K. Muthukrishnan, & others, 2015)[*27,28], The most generally used Na-CMC has degree of substitution of 0.7. The Acetone is colorless high volatile and reactive

compound and it also have an flammable capacity. The widely used solvent for synthetic fibers plastics for extracting regular bio-marker also find the application in cosmetics, petro chemical industries(X. Li & others, 2012)*[29].

The low concentration it is non-toxic, non-carcinogen and non-neurotoxic. However concentration of acetone is above 10,000ppm in atmosphere it causes irritations to eye and throat and also cause nausea (X. Li & others ,2012)*[29,30]. The human breath analysis is a rapid and non-Invasive method used in bio-maker for various diseases (M. Righettoni & others ,2010)*[31]. The acetone hydro carbon, ammonia are some of them have endro-carbon, ammonia are some of them have endogenous molecules present in human breath by change the high concentration it indicated various diseases (W. Miekisch & others ,2004)*[32] sulphide present content in human breath indicates oral (A.D. Amico & others ,2008)*[33] Amonia concentration content renal failure(S. Davies & others ,1997)*[34]

The acetone is found in the breath of diabetic patients (T. Do & others ,2012)*[35] ,The evolution of Nitric oxide indicate presence of asthma (E. Heffler & others ,2006)*[36], The most of the human are affected by diabetes for diabetic patients, fat is utilized as energy for cellulose rather than glucose due to absence of insulin. Hence keto-acidosis occur due to ketone accumulation during metabolic activity to odour of acetone in breath (D. Guo & others 2010)*[37]. The concentration of acetone in breath exceeds 2ppm. When the person have diabetes where as in less 1ppm for healthy person (P. Gao & others ,2012)*[38]. The researcher of his research work towards fabrication of Zinc Oxide thin film they could noticed that acetone vapors in room temperature.

II. Experimental section:

The Researcher could start the process by mixing two different solution, they are namely Originated and Thickening agent ,to following the preparation:

a) preparation of Originated solution:

At first the homogeneous solution were dissolving in 4.68gms of Zinc nitrate hexa hydrate in 100ml of di-water, for preparing originated solution. That both mixing solution act as an originated solution.

b) For preparing thickening agent:

Further, 2gms of Na-CMC is slowly dissolved in 100ml of di-water ,This dissolved solution act as a thickening agents. The Na-CMC take time for dilute because the Na-CMC powder could be like a cotton ball flow on water so we can slowly but Na-CMC on di-water and dissolve by using steering method. After that, the Originated solution was added slowly to thickening agent with drop-by-drop 1ml /min. The solution color was changing up to when 8ml of Originated solution was added ,when we see that the nano particle was split and the bubbles are forming from that solution .

During the entire process that the solution was stirring at constant and the temperature are also maintained at 70^{o}c (+,- 2^{o}c). And finally the white Viscous solution is obtained. That solution look like white jelly form. The impurities could be removed by washing the gel with di-water and repeat this process for 6-7 times or removing impurities.

The washed was kept in constant stirring for 24 hours ,after that we use that gel solution for dip coating. For dip coating the researcher used glass substrate which was cleaned by adding chromic acid +100ml of di-water for 30 min on beaker , then cleaned that substrate with Acetone and the solution of sodium hydroxide pellet for 20min over the beaker and then again clean the substrate with Acetone after that process completed put the substrate with iso-prabal alcohol through beaker to ultrasonically cleaned for 30min .The thin film of ZnO were obtained by immersing substrate in sol gel dip coating for 30min vertically with following table .

TABLE (1): DIP COATING MACHION SLIDE SETUP PROCESS

SNO	WORKING SETUPS	TIME/SIZE
1	STARTING POSITION	30mm
2	DIP LENGTH	120mm
3	DIP SPEED	3mm/sec
4	RETURN SPEED	9mm/sec
5	DIP DURATION	30sec
6	DRY DURATION	1min
7	NUMBER OF DIPS	10 dips

The above mentioned process are Repeated for 10 times for uniform coating and dry at 120^{o}c for 10 min .And its taken for annealing at 450^{o}c for 3 hours to get crystalline ZnO nano – particles on thin films. The

main purpose of using Na-CMC is to get tailored nano-particles and provide sticking together of ZnO nano particles on natural glass substrate.

III. Characterization

X-Ray diffractometry and field emission secondary electron micrograph (FE-SEM) has been characterized were done to conclude crystallinity, crystallographic structure and surface morphology in ZnO thin film annealed at 450^{o}c. The XRD pattern were achieved by X-ray power diffractometer equipped with cu kα radiation have wave length of 0.1548nm as a basis. The crystal size was calculated using debye scherrers formula (S. Suwanboon & others ,2011)*[39].

$D = K\lambda / \beta COS\theta$

Where k is shape factor (k=0.89), λ is the wave length of X-ray source, βis full wave length at half maximum (FWHM) and θ is the diffraction angle morphological investigation were done using FE-SEM . The gas sensing performance of the film erer studied by using chemiresistive method .

Iv. Result and discussion :

4) a. CHARACTERISATION OF THIN FILMS:

The crystalline character of dip coated thin films was characterized by XRD fig:1 shows. The XRD pattern, were they characteristic peaks of ZnO at {100},{002} plane respectively, from the presence of hexagonal heart in shape nano structure was conformed by using {100} plane and peak broadening concludes the formation of Nano- particles. The size of the film was measured by using equ (1) and it found 20 nm.

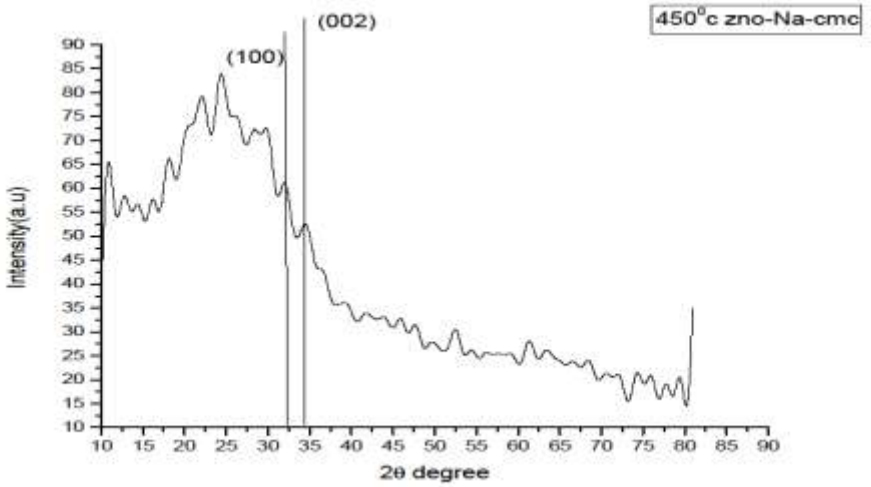

Fig: (1).THE RESULT OF X-RAY DIFFRACTION:

The thin layer of film coated on glass substrate, have an strong bulge was seen in XRD-pattern of ZnO thin films. FE-SEM of thin film shown a heart in shape Nano structure has been shown in fig.2 Through over all morphology been similar to un even and irregular.

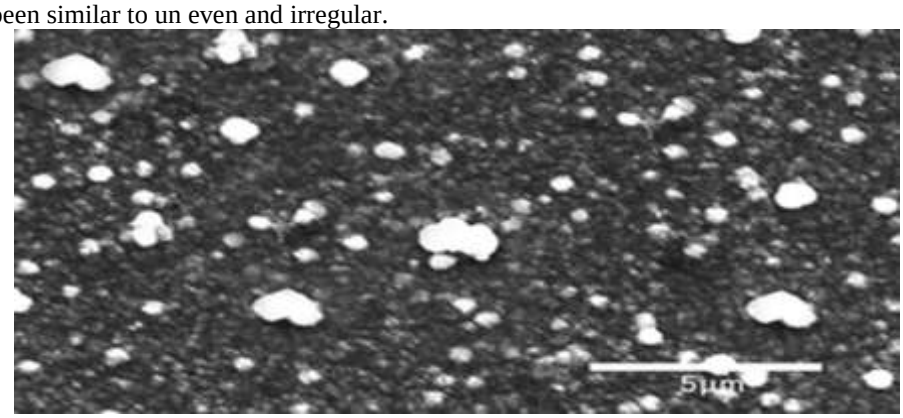

Fig.(2) RESULT OF FIELD EMISSION SCANNING ELECTRON MICROSCOPY:

4) b. GAS SENSING STUDIES:

According to my research that the research could be studied the gas sensor by using No_2 gas that has been have sensing property of variation of resistance. The thin film has been exposed in air, atmospheric oxygen get adsorbed on the film surface in any one of the form (o^2,o,o_2^-) (Y. Zeng & others ,2009) *[40]. It leads to increase in height of the potential barrier and its causes on increase in resistance of the material. The point at which the resistance remain almost stable is considered as base resistance(P. Dhivya & others ,2014) *[41,42]. When the researcher could given his samples for studies that they testing SNO_2 sensors. The gas sensor based on SNO_2 exhibited to the p-type response to acetone . They have super acetone sensing property can be qualified.
The responses of sensor are based on ZnO 1*105ppm acetone as a function of operating temperature while for reducing gas trapped electrons are realized back to the film surface the resistive film decreases for base resistive (P. Rai & others ,2012)*[43,44].

4) c. THE MACHANISM AND GAS SENSING PROPERTIES:

The researcher shown the gas sensor based on ZnO has a maximum gas reaction at the operating temperature of 280^OC. while the sensor based on ZnO shows higher responses at 310^oc. As the researcher shows the result 280^oc respectively selected an operating temperature for ZnO sensor in the following testing method .The researcher main result of gas sensor has been shown in the sensed the gas, that the below tabulation has been verified that.

Tabulation of gas sensor result:

Prec	CVD method	Tdep ^oc	Form	Features nm	Sensor type	Top ^oc	Ppm	Gas	R	Tres
Zno	CVD	450	Film	380L	Ω +0 SNO	280	1×105	NO2	1.38	-

Prec – precursors,
Tdep – temperature of deposition ,
Top - operating temperature,
Tres – response time,
Ppm – part per million,
R = Ra / Rg (oxidative gas),
R = Rg / Ra (reduction gas).
From the above tabulation that the researcher could take an constant higher value for this sensor method. That the required time will be constant at higher value The ratio between change in resistance from the base resistance is response to sensing material given below (P. Shankar & others ,2013)*[45].
For reducing gas s=Ra/Rg
For Oxidizing gas s=Rg/Ra
Where Ra is resistance of the film in air and Rg is the resistance of the film in presence of test gas. In the researcher work that the sensor response in teat gases such as acetone, ethanol . The computer dynamic gas sensing setup was used in same one of paper (G.K. Mani & others ,2013) *[46]. The response of film towards 1×105 ppm of acetone, ethanol, etc. These variation in magnitude of response arises from the amount of chemisorbed oxygen on the surface of the film and the variation in interaction strength of the test gas with the surface of sensing element (D. Sivalingam & others ,2012)*[47].

THE ACETONE SENSING MACHENISM:

The sample could be test with the closed chamber of 50ppm acetone injected, due to the reducing nature, acetone tends to release the trapped on ZnO thin film surface. The decrease in the high potential barrier so conduction increased. This causes resistance to fall from base resistance. This possible chemical for acetone is as follows (S.S. Nath & others ,2010)[48].

From the Researcher observation that lower detection limit of annealed ZnO thin film was 105ppm and its response was found s=1.38 very low response 1ppm was due to very few acetone molecules for interaction. The very high response of 1×105ppm . The reason for this proportionally between acetone concentration response was catalytic effect of ZnO Nano sphere and participation of more acetone molecules with adsorbed oxygen molecules(R. Pandeeswari & others ,2014)*[50].

V. Conclusion

The zno thin film sol-gel dip coating method and its gas sensing characteristics were studied. The most stable heart in shape nano structure was conformed with XRD. The sensor test was done by closed chamber at room temperature . The test shown that ZnO thin film can act as a acetone sensor for an selectivity of constant values. The limit of room temperature lower detection of zno thin film observed to 1×105ppm of acetone with the response of 1.38. Hence the Researcher could shown that it has identified diabetes.

Referance

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