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Synthesis, Characterization and Development of Opto-Electronic Humidity Sensor using Copper Oxide Thin Film

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Abstract: The present paper reports the study of modulation in light transmitted through nanostructured cupric oxide thin films with the exposure of moisture at room temperature. For this purpose the precursor of CuO was prepared and was then investigated using SEM and UV-visible absorption techniques. SEM showed the porous nature of the material and energy band-gaps were estimated as 2.8-3.2 eV for different concentrations by UV-Vis spectrophotometer. The precursor of CuO in the form of gel was prepared and later used for the deposition of thin film on borosilicate substrates with dimensions 1.5×1.5 cm^2 using sol-gel spin coating technique. The film was employed as transmission based opto-electronic humidity sensor. Maximum sensitivity was found 0.85 µW/%RH. As the investigated opto-electronic sensor has remote access capability, therefore, may be used to replace an electrical humidity sensor.

Keywords: Nanostructure, opto-electronic humidity sensor, sol-gel spin coating, cupric oxide.

I. INTRODUCTION

In recent years advancements in sensor manufacturing technologies have driven by post-process high-speed, low-power and low-cost microelectronic hybrid circuits [1-3], modern signal conditioning methods and progress in miniaturization technologies [4-5] product reliability. Furthermore, it is vital to know the degree of efficiency of each sensor related to its calibration circumstances and sensing mechanism [6-7]. Today, simulation techniques and design aids are adequately used to predict and improve output data prior to implementation of mass production processes to save time and enhance quality [10-11]. Miniaturization of sensor devices offers numerous advantages such as low hysteresis [5], batch fabrication [12], and ease of packaging/integration along with the corresponding cost reductions [13-16].

It is well documented that humidity plays a significant role in every part of the Earth including biology and automated industrial processes. To have a desirable surrounding atmosphere, it is essential to monitor, detect and control the ambient humidity under different conditions ranging from low temperature to high or in mixtures with other gases by precise and provident sensors [17-18]. Utilization in intelligent systems and networks as monitoring sensors to determine the soil moisture during irrigation in agriculture, or for diagnosis of corrosion and erosion in infrastructures and civil engineering are among the major applications of humidity sensors [19]. In fact, the need for protection of environmental conditions has been leading to extensions in various humidity sensor developments based on the use of physical and chemical methods in presence of organic, inorganic or hybrid materials since long back [20-21]. Advancement of humidity sensor systems encompasses enhanced efforts in betterment of transducer performance such as sensing elements [22], structure design [23-24], principle of mechanism [25-26], and fabrication technologies [27]. In this milieu, the transducer materials are the key features, followed by the availability of suitable manufacturing technologies, free choice of device geometrical properties to attain the required dimensional efficiencies, optimization of surface for the occurrence of conductance, ease of production flow and investment expenses.

Copper (II) oxide or cupric oxide (CuO) is the higher oxide of copper. As a mineral, it is known as tenorite. It is a black solid with an ionic structure which melts above 1200°C with some loss of oxygen. Copper (II) oxide belongs to the monoclinic crystal system, with a crystallographic point group of 2/m or C2h. The space group of its unit cell is C2/c, and its lattice parameters are a = 4.6837(5), b = 3.4226(5), c = 5.1288(6), α = 90°, β = 99.54(1)°, γ = 90°. The copper atom is coordinated by 4 oxygen atoms in an approximately square planar configuration [28].

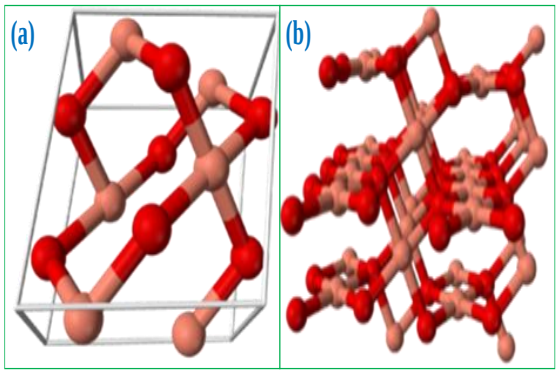

Fig. 1 CuO (a) Unit cell (b) Crystal structure

CuO has molar mass 79.545 g/mol and density 6.315 g/cm^3. Its melting point and boiling points are 1326 °C and

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2000 °C. It is insoluble in water, slight soluble in alcohol or ammonia solution but soluble in dilute acids e.g. NH_4Cl, $(NH_4)_2CO_3$ and potassium cyanide. Under high temperature, copper oxide meets with hydrogen or carbon monoxide and can restore copper metal. Nano-structured copper oxide is extensively used as catalyst, superconducting materials, thermoelectric materials, sensing materials, glass, ceramics and other fields [29-31]. In addition, it can be used as rocket propellant combustion catalyst. It not only can significantly improve the homogeneous propellant burning rate, lower pressure index, but also can better perform as the catalyst for the ammonium per chlorate composite propellant. There are more uses of cupric oxide such as in ceramic resistors, gas sensors, humidity sensors, magnetic storage media, near-infrared tilters, photoconductive and photo-thermal applications, semiconductors, solar energy transformation and high-tech superconductors [31].

Previously, a resistive humidity sensor was successfully fabricated based on hydrothermally synthesized coral-like CuO nanostructures by selective growth on ZnO nanorods (NR) at low temperatures [32]. Heterojunction p-n diodes fabricated from the CuO/ZnO nanocorals (NC) revealed the stable and high rectification diode properties with a turn-on voltage of ~1.52 V and negligible reverse current. The humidity sensing characteristics of the CuO/ZnO NC diodes exhibit a remarkable linear (in a semi-logarithmic scale) decrease in the DC resistance by more than three orders when the relative humidity is changed from 30-90 %. The NC humidity sensor was also found to reveal the highest sensitivity factor ~ 6045 among available data for the constituent materials and a response and recovery time of 6 s and 7 s respectively. Also N. Serin et al have reported the growth of CuO nanowires on an oxidized Cu wire and the fabrication of a CuO nanowire humidity sensor. It was found that they could transform a Cu wire into CuO/Cu_2O/Cu core-shell tri-layers covered with high density CuO nanowires by thermal annealing. It was also found that steady state currents of the sensor were about 2.44, 2.32, 2.23 and 2.15 μA, respectively, when measured with 20, 40, 60 and 80 %RH. Furthermore, it was found that sensing property of the fabricated device was stable and reproducible [33]. But it was based on the measurements of electrical parameters which do not have remote access capability, therefore, the preparation of nanostructured cupric oxide thin film and its application as opto-electronic humidity sensor was planned.

II. EXPERIMENTAL DETAILS

A. Synthesis of material

The sol-gel synthesis process is one of the most common processing routes for the synthesis of a wide variety of materials in desired shapes, like particles, fibers and thin films. A sol is a stable dispersion of colloidal particles or polymers in a solvent. The particles may be amorphous or crystalline. An aerosol is particles in a gas phase, while a sol is particles in a liquid, a gel consists of a three dimensional continuous network, which encloses a liquid phase.

The choice was made taking into account the fact that hydrolysis of acetate group gives products which are soluble in the solvent medium and get easily decomposed into volatile compounds under the heat treatment. A colloidal solution of copper acetate in ethanol was used as precursor. Copper acetate added to ethanol (10 g dm^{-3}) was stirred using magnetic stirrer for 3 h. The resulting solution was sonicated for 2 h and kept overnight for stabilization. Bluish precipitate was obtained which was filtered and dried in oven for 1 h. Resultant powder was ground and kept inside an electric furnace at 450˚ C for 2 h. Brown-black coloured pure CuO powder was obtained.

B. Preparation of thin films

Gel was prepared by mixing CuO powder in ethanol and then stirring using magnetic stirrer. Spin coating was used for the deposition of thin films on glass substrates. A small puddle of the material was placed onto the centre of a substrate and was then spinned at high speed of 3000 rpm. Films were prepared on glass substrates which were then heated on a hot plate at 60˚C for 15 min.

III. CHARACTERIZATION

A. Scanning Electron Microscopy

Surface morphological investigations of the sensing material were carried out using Scanning Electron Microscope (JEOL, JSEM-6490LV). Pores can be visualized in the photograph shown in Fig. 2. Surface morphology of the film exhibits the porous structure which is quite favourable for the adsorption process. These pores/active centres serve as humidity adsorption sites and the sensitivity of the sensor depends on the size of these pores. Accordingly, macroporous CuO with a higher surface area provides more surface adsorption sites, which is advantageous to the sensor operating at room temperature. The sensing property is based on the moisture adsorption at the surface of the material, which causes a change in the output power due to the change in refractive index (r.i.) of the material. The surface of such morphology has many dangling bonds on account of which the sensing surface is optically very reactive. Owing to this reactivity, the sensing surface easily adsorbs the moisture that comes into the contact and increases the r.i. of the film.

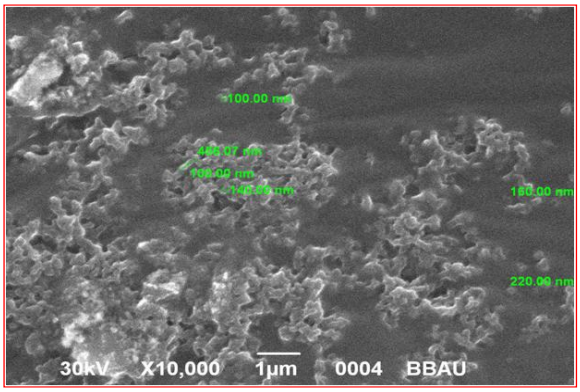

Fig.2. SEM photograph of CuO

B. UV-visible (UV-Vis) absorption analysis

Optical characterization of the sensing element was carried out using the UV-visible spectrophotometer in the range

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200 nm-1100 nm. The UV-Vis photospectra of solution of CuO nanopowder in ethanol was recorded with respect to ethanol placed in the reference beam using single beam spectrophotometer. The spectra were analyzed [34] by plotting $(\alpha h\nu)^2$ vs hν, based on eq.

$$\alpha h\nu = A\,(h\nu - E_g)^{n/2}$$

Where, α is absorption coefficient, A is a constant and n is the exponent that depends upon the quantum selection rules for the particular material. Here the value of n is taken as ½ showing the direct allowed type transition. A straight line is obtained when $(\alpha h\nu)^2$ is plotted against photon energy (hν), which indicates that the absorption edge is due to a direct allowed transition. The intercept of the straight line on hν axis corresponds to the optical band gap (E_g). The absorption spectra and optical band gap for different concentrations are shown in Fig. 3 and 4 respectively.

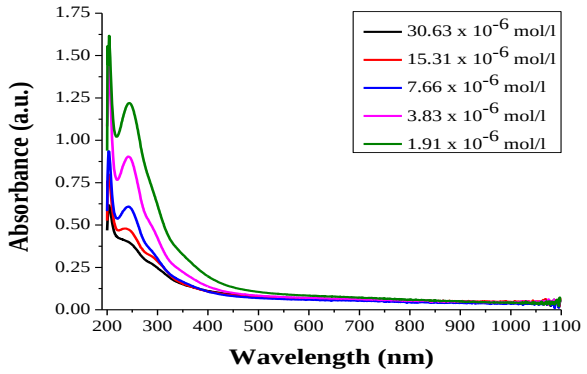

Fig. 3 Plot of absorption vs. wavelength for CuO-ethanol solution at different concentrations.

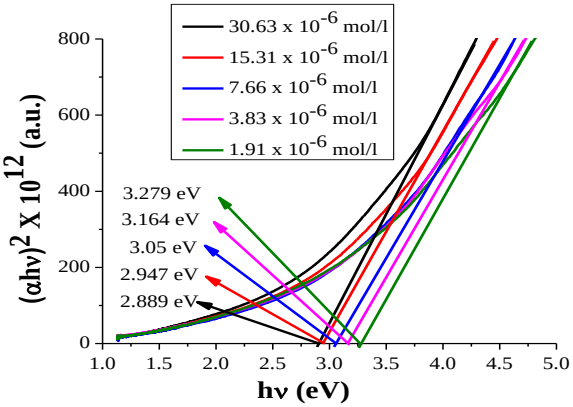

Fig. 4 Plot of $(\alpha h\nu)^2$ vs. hν for CuO - ethanol solution at different concentrations

Concentration (mol/l)	Optical Band gap (eV)
30.63 x 10^{-6}	2.89
15.31 x 10^{-6}	2.947
7.66 x 10^{-6}	3.05
3.83 x 10^{-6}	3.164
1.91 x 10^{-6}	3.279

Table I: Variation of optical band gap for different concentrations.

Variations of optical band gap for different concentrations are depicted in Table1 and it can be observed that as the concentration decreases the optical band gap increases.

IV. DEVICE ASSEMBLY

Fig.5 represents the schematic layout of the device [35] used. Humidity controlled chamber was designed in our laboratory using transparent glass, inside which hygrometer and humidifier/dehumidifier dish were placed and the film was fixed on the opposite wall of the chamber. A 2 mW He-Ne laser was used as input light source, the beam of which was expanded using beam expander having power 10X and allowed to fall on the whole surface of the sensing film. The light beam coming out from the humidity chamber was converged through a lens combination to an optical power meter. The change in light intensity with respect to change in relative humidity inside the chamber was then measured for an interval of 5 %RH. Saturated aqueous solution of K_2SO_4 was used as humidifier for increasing the %RH from 10 to 90 whereas saturated aqueous solution of KOH was used as dehumidifier to decrease the humidity inside the chamber from 90 to 10 %RH.

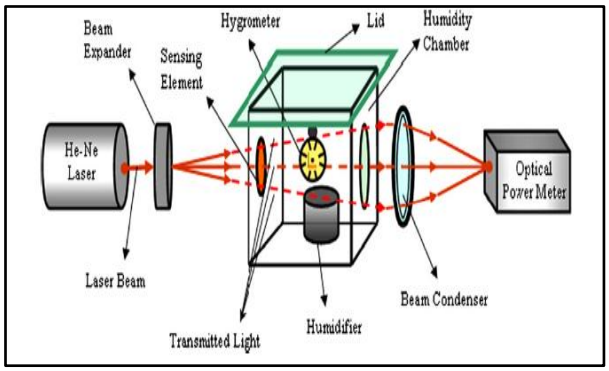

Fig.5 Device assembly for the study of opto-electronic humidity sensing

V. OBSERVATION

Fig. 6 shows the response of the humidity sensing film with the change in %RH inside the humidity chamber. Curve 'a' shows the linear decrease in transmitted power with increase in %RH; however, the Curve 'b' shows the increase in transmitted power with the decrease in %RH. While performing the decreasing mode of %RH, the ending values of optical powers were found to be reduced. Sensitivity of sensor was calculated using the following formula[36]:

$$\text{Sensitivity} = \frac{\text{Change in Output power}}{\text{Corresponding change in \%RH}}$$

$$S = \frac{\Delta I_t}{\Delta \%RH}\ \mu W / \%RH$$

The sensitivity obained here for increasing mode of %RH is 0.85μW/%RH and the hysteresis obtained is 15%.

VI. RESULT & DISCUSSION

The humidity sensing characteristics of CuO based sensing film deposited on a plane transparent borosilicate glass

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substrate were studied and variations in optical transmitted power with the variation of %RH were recorded. The corresponding data were plotted in Fig. 6. The modulation in the light intensity transmitted out from the sensing film was mainly due to the variation in refractive index and thickness of the film with the variation in %RH. Depending upon the surface morphology, the sensing mechanism is based on adsorption of water vapor and capillary condensation as shown in Fig.7 [37]. The range of %RH can be divided into three regions viz. dry (below 40%RH), medium (40-70%RH) and very humid (above 70%RH). Initially the cupric oxide film was free from water molecules, having only dry air in its pores. When exposed to low humidity, adsorption of water vapor takes place rapidly on the surface of film. Due to highly electropositive nature of metal ion at metal oxide surface of the sensing element, it has tendency to break one of the O-H bonds of H_2O to form a strong chemical bond between M^+ and OH^- . Thus initial layer is chemisorbed and in this layer output power starts decreasing due to the bending of light. As the %RH increases, adsorption on the surface increases. However, in mid humid region of 40-70 %RH, adsorption takes place on the surface as well as on the walls of capillaries, leading to a further decrease in the output power at detector. In very humid region the condensation of water vapor takes place through the capillaries and forms a meniscus in the capillaries of the film. Therefore in this region, the output power decreases stridently. Moisture detection would depend significantly on the porosity and thickness of the deposited film, as the extent of adsorption of water vapor causes the increase in refractive index of the film.

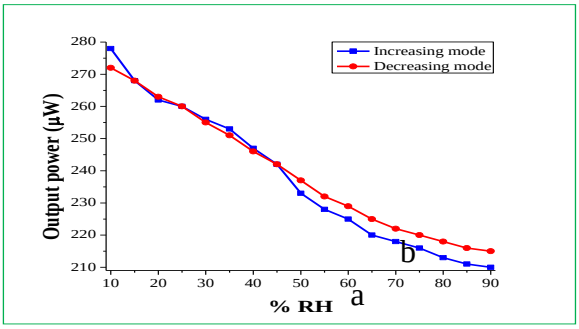

Fig. 6 Variation of the output power with increasing and decreasing %RH

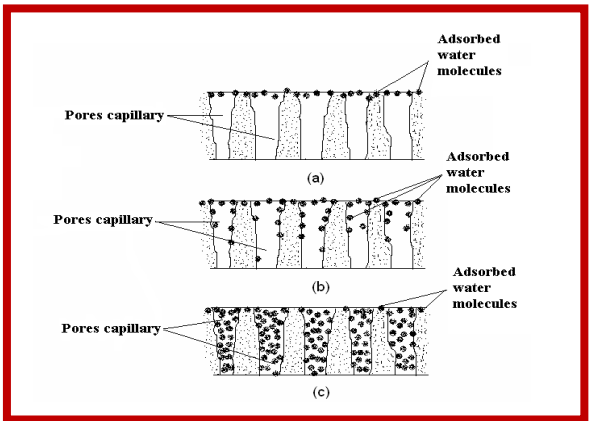

Fig. 7 Water adsorption mechanism on the film surface

VII. CONCLUSION

The present study, thus, leads to the conclusion that CuO thin films prepared by sol-gel spin coating technique can be employed as an optoelectronic humidity sensor based on the modulation in output power of light transmitted through the sensing element. SEM exhibited that the material being porous had sufficient adsorption sites to attract the moisture. Optical band gap of the material measured by UV-Vis spectrophotometer was in the range of 2.8-3.2 eV. The sensitivity of sensor was found as 0.85 μW/%RH. The remote access capability of such type of sensor based on CuO nanoparticles would be very useful for futuristic development of humidity sensor. Work is under investigation.

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BIOGRAPHIES

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Ms. Samiksha Sikarwar is pursuing Ph.D. in Babasa-heb Bhimrao Ambedkar University, Lucknow, U.P., India. Her research area of interest includes the synthesis of multi metallic oxide nanoparticles and their applications as opto-electronic humidity sensor. Currently she is an INSPIRE fellow, sponsored by DST, Govt. of India. She has been also been awarded the certificate of extra-ordinary talent in All India Talent Search Examination organized by the Children's Education Trust of India in 2002.

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