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Copper Nickel Oxide Thin Films Deposited by DC Sputtering for gas sensing applications

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Abstract: Copper nickel oxide ($CuNiO_2$) thin films were deposited onto unheated glass substrates by DC reactive magnetron sputtering the composite target of $Cu_{50}Ni_{50}$ at a fixed oxygen partial pressure of $3x10^{-4}$ mbar and sputter pressure of $3x10^{-2}$ mbar. The as-deposited copper nickel oxide films were annealed in air at temperature of 200°C in one hour. The as-deposited and annealed film was characterized for their chemical composition, electrical, optical and Hydrogen gas sensing properties. The as-deposited films were amorphous in nature. The electrical resistivity of the film increased with annealing temperature due to the filling of oxygen ion vacancies. The films annealed at 200°C exhibited the crystallite size of 25 nm, electrical resistivity of 12 Ωcm and optical band gap of 1.97 eV.

Keywords: $CuNiO_2$ thin films, DC magnetron sputtering, Electrical, Optical and Hydrogen gas sensing properties.

I. INTRODUCTION

Hydrogen is a good alternative to alternative clean energy fuels. In this case, large-scale hydrogen production, distribution and combustion of engines will require advanced control systems to ensure the safe storage and use of gases. Therefore, new smart sensors with high H_2 sensitivity will be needed. A lot of work has been done to find suitable materials for hydrogen sensors, especially materials such as metal oxides SnO_2 [1], In_2O_3 [2], ZnO [3], CuO [4] and NiO [5] films were investigated as sensitive thin film resistors. A method for detecting Hydrogen in the air using NiO films has also been reported almost successfully [6], but real sensors need to optimize the detection surface to ensure high sensitivity, short response time, good repeatability and reliability. In this work, we focus on the characteristics of thin films of $CuNiO_2$, with the aim of developing a sensor capable of detecting concentration of hydrogen in the closed chamber with 5500 ppm at room temperature.

II. EXPERIMENTAL

DC-reactive magnetron sputtering method was used to deposit a thin film of copper nickel oxide ($CuNiO_2$) onto a clean glass substrate by sputtering an equimolar copper-nickel $Cu_{50}Ni_{50}$ target with a diameter of 50 mm and a thickness of 3 mm. The sputtering chamber is evacuated using a combination of a diffusion pump and a rotary pump to reach a limit pressure of $5x10^{-5}$ mbar. Pure oxygen and argon are used as reaction gas and sputtering gas, respectively, and enter the sputtering chamber through a fine controlled needle valve. The $CuNiO_2$ film was deposited on the substrate that was kept at room temperature in a pulverized manner downwards, with a fixed partial pressure of oxygen of $3x10^{-4}$ mbar, a sputtering pressure of $3x10^{-2}$ mbar and a sputter power of 90 watts.

The Veeco Dektak depth target profilometer was used to measure the thickness of the film after deposition and annealing. Energy dispersion X-ray analysis (INCA Penta FETX3) connected to a scanning electron microscope (Carl Zeiss EVO MAIS model) is used to determine the chemical composition of the film. An X-ray diffractometer (Rigaku Mini Flex II) was used to analyze the crystal structure of the film using a single-color copper CuKα radiation with a wavelength of 0.15406 nm. The surface morphology was analyzed with an atomic force microscope (Seiko Instruments model SPA 400). The resistivity of the film was measured using four probe technique. To determine the optical band interval, a UV-Vis-NIR double beam spectrophotometer was used to record the transmittance of the film in a wavelength range of 300-1000 nm.

The dynamic measurement of the sensor response is done through a metal chamber at a temperature of 30 ° C under a controlled flow of H_2 and dry air. A Keithley 485 peak ammeter was used to record the current through the real-time $CuNiO_2$ sample at a polarization voltage of 1 V. Monitor current changes at fixed hydrogen the concentration of 5500 ppm for the as-deposited and annealed film. The response of the sensor S is calculated as the ratio of (I_g-I_{air} / I_{air}), where I_g and I_{air} are the current of the sensor under the flow of Hydrogen with and without Hydrogen, respectively.

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III. RESULTS AND DISCUSSION

The thickness of the deposited copper-nickel oxide film was 130 nm. The deposited film was annealed in air at 200 °C for 1 hour. Figure 1 shows a dispersive X-ray spectrum of energy representative of the as deposited film. The EDAX spectrum shows characteristic peaks of copper, nickel and oxygen. The deposited film showed a compositional content of copper = 26.6 at. %, Nickel = 25.5 a. % and oxygen = 47.9at. %.

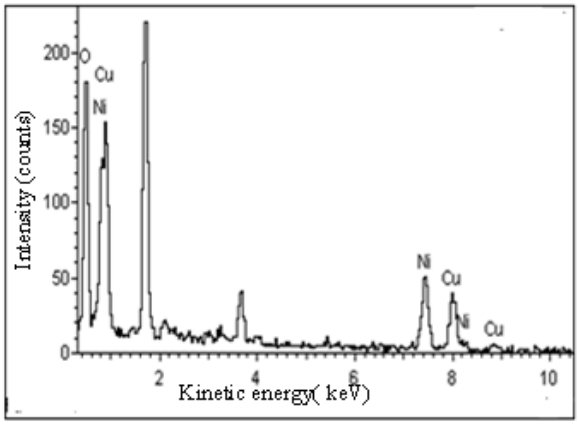

Figure. 1 Energy dispersive X-ray spectrum of as-deposited $CuNiO_2$ film

As annealing temperature increased the content of oxygen increased that is at 200°C the films contained the copper = 25.7 at. %, nickel = 24.5 at. %, and oxygen = 49.8 at. %. As the annealing temperature increased the oxygen ion vacancies were filled hence lead to the formation of $CuNiO_2$ [7].

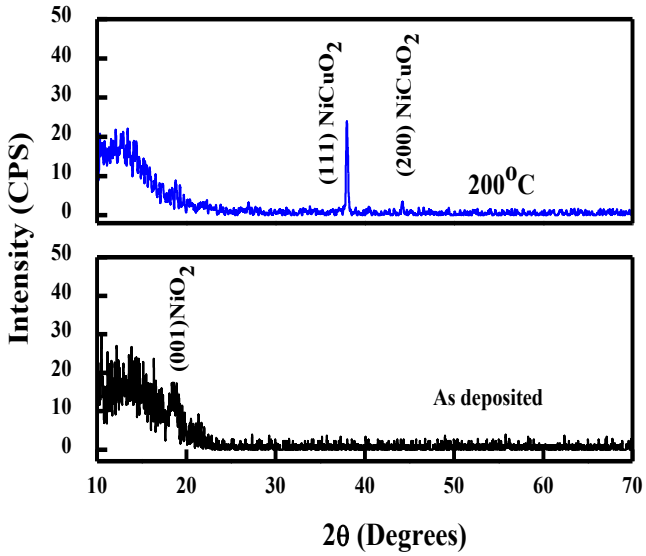

Figure. 2 X-ray diffraction profiles of as-deposited and annealed $CuNiO_2$ films at 200°C.

X-ray diffraction profiles (XRD) of as-deposited and the $CuNiO_2$ films annealed in air at different temperatures are shown in figure 2. The as-deposited films were X-ray amorphous. It contains a weak (001) reflection of NiO_2. The films annealed at temperature of 200°C were polycrystalline in nature. The diffraction peak located at 37.04° was related to (111) reflection and a weak peak observed at 43.8°correspond to (200) reflection of $CuNiO_2$. The observed diffraction reflections were in accordance with the JCPDS data of $CuNiO_2$ [8]. The crystallite size of the films was calculated from the full width at half maximum intensity of X-ray diffraction (111) reflection by using Debye-Scherrer's relation [9]. Crystallite size of the films increased from 25 nm at an annealing temperature of 200°C. It clearly indicated that the annealed film was of nanocrystalline $CuNiO_2$ [7].

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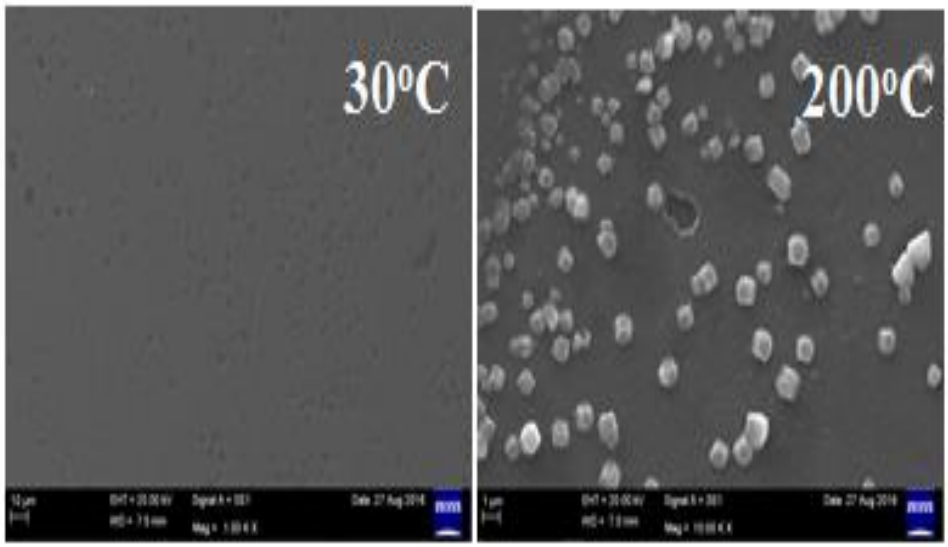

Figure. 3a Two and three dimensional SEM images of $CuNiO_2$ films: (a) As-deposited and the film annealed at (b) 200°C.

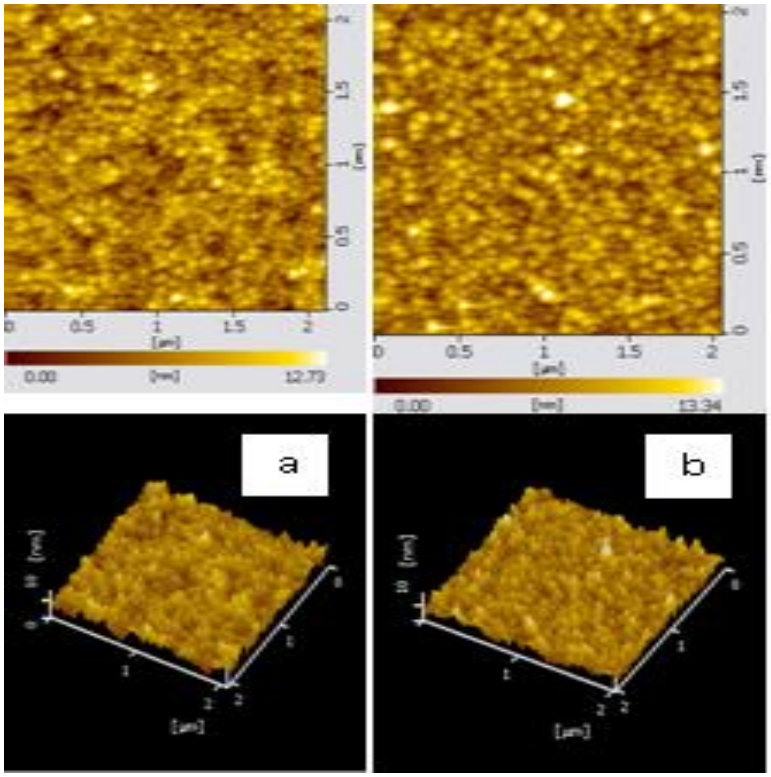

Figure. 3b Two and three dimensional AFM images of $CuNiO_2$ films: (a) As-deposited and the film annealed at (b) 200°C.

Surface morphology of the films was analyzed with atomic force microscope. Figure 3 shows three and two dimensional atomic force micrographs of as-deposited and the films annealed at 200°C. It is seen from the micrographs that the grown grains were of uniform in all the films. The as-deposited film was of fine grain structure due to amorphous nature (figure 3a). As the annealing temperature increased the size of the grains increased and grown grains were almost in spherical shape (figure 3b). The grain size of the films increased from 56 nm to 100 nm in the as-deposited and the films annealed at 200°C. The root mean square roughness of the as-deposited films was 2 nm and it increased to **12** nm in the case of films annealed at 200°C. Low roughness in the case of annealed film was due to the small sized grains presence in the sample [7].

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The electrical resistivity of the films was influenced by the annealing temperature. The as-deposited film exhibited the resistivity was 0.47 Ωcm. It is clearly seen that as the annealing temperature increased the electrical resistivity of the films increased. The films annealed at 300°C exhibited the resistivity of 22 Ωcm. Increase in the resistivity with annealing temperature was due to filling the oxygen ion vacancies [7]. The electrical resistivity reported were $3x10^4$ Ωcm [10] and 67 Ωcm [11] in RF magnetron sputtered $CuNiO_2$ films.

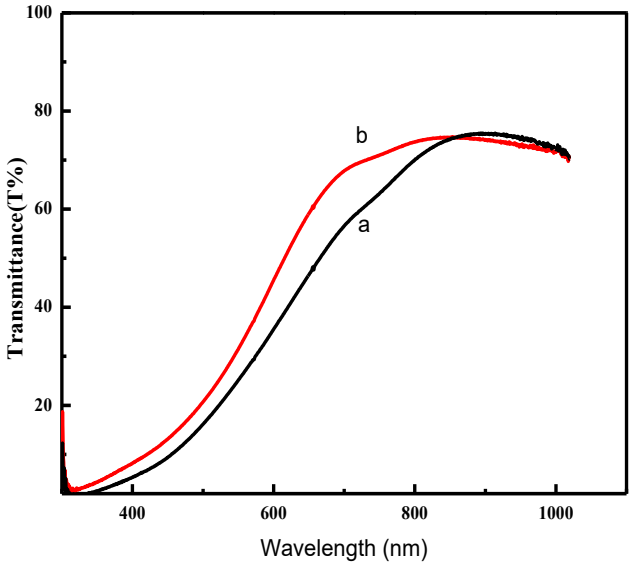

Figure. 4 Optical transmittance spectra of copper nickel oxide films at 30°C and (b) annealed at 200°C.

The wavelength dependent optical transmittance spectra of $CuNiO_2$ films annealed at different temperatures are given in the figure 4. It is seen from the spectra that the optical transmittance of the films increased with increase of annealing temperature. Low transmittance in the as-deposited films was due to the presence of oxygen ion vacancies which scatter the light photons by metallic species hence reduction in the transmittance. The optical absorption coefficient of the films was calculated from the transmittance. Optical band gap of the films was evaluated from the absorption coefficient using the Tauc's plots [12]. The plots of $(\alpha h\upsilon)^2$ versus photon energy ($h\upsilon$) of the films are shown in the figure 5.

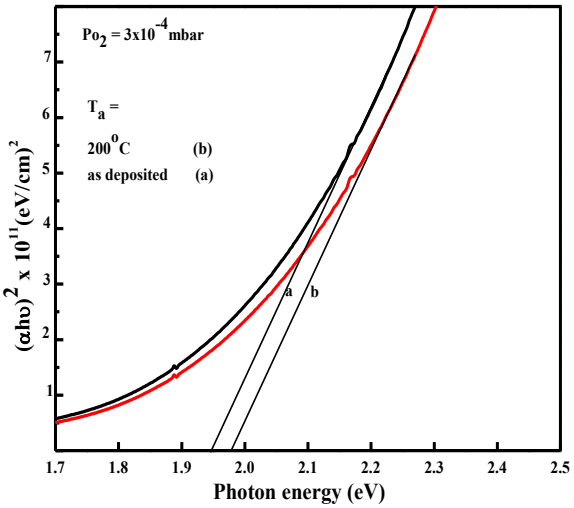

Figure. 5 Plots of $(\alpha h\upsilon)^2$ versus photon energy ($h\upsilon$) of copper nickel oxidefilms.

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Extrapolation of the linear portion of the plots leads to the optical band gap of the films. The optical band gap of the as-deposited films was 2.15 eV. As the annealing temperature increased to 300°C the optical band gap of the films increased to 2.06 eV [7].

IV. HYDROGEN GAS SENSING PROPERTIES

The mass flow meters/controllers used to supply a constant flow (5000 ppm) during test measurements. In this case, the resulted concentration of H_2 gas was computed by the following eq. 1. The relative sensitivity (S%) could be written in terms of the electric current generated in the semiconductor-based gas sensor on the application of a constant bias voltage [13].

$$S\,\% = \frac{I(g)-I(air)}{I(air)} x100 \quad \text{--------} \quad (1)$$

Where Ig is the current measured in the presence of the gas being sensed and I_{air} is the current measured in air ambient. Figure 6 shows the response-recovery characteristics of $CuNiO_2$-based at 5500 ppm hydrogen at 30°C and 200°C. The response and recovery time of the $CuNiO_2$ sensor observed at 5500 ppm of hydrogen was found to be 10 and 4s. The annealed $CuNiO_2$ is 8 s and 2 s, respectively. It is clear from figure 7 that the response of the NiO sensor increases after the annealing temperature. The gas detection mechanism of $CuNiO_2$-based sensors implies a change in current, which is caused by the adsorption and desorption of oxygen molecules on the surface of thin films. The reaction mechanism can be expressed according to the following equation

$$O_2\,(ads) + 2e^- \rightarrow 2O^- \quad \text{----------} \quad (2)$$

$$2H_2(ads) + 2O \rightarrow 2H_2O + 2e \quad \text{----------} \quad (3)$$

When a $CuNiO_2$ based sensor is placed in the air, oxygen in the atmosphere captures the electrons in the valence band and ionizes, resulting in the formation of layer of hole accumulation [14]. Since holes are the main transporters in $CuNiO_2$, the resistance of $CuNiO_2$ decreases in the

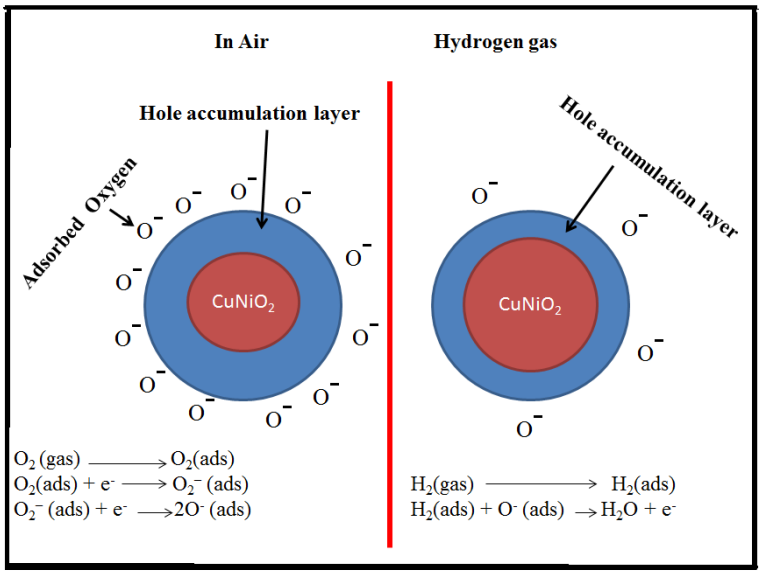

Figure. 6 Shows a schematic diagram of the CuNiO2 gas detection mechanism in air and hydrogen.

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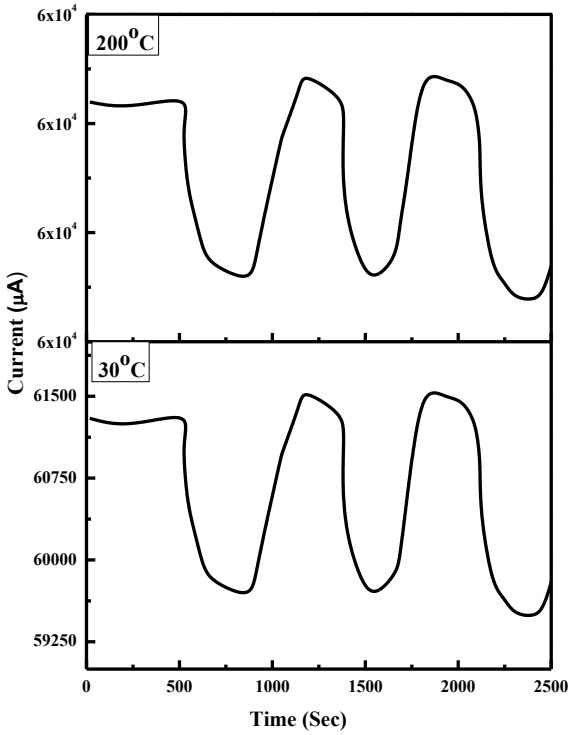

Figure. 7 Schematic diagram of gas sensing Mechanism of $CuNiO_2$

presence of air. When a $CuNiO_2$ based gas sensor is exposed to a reducing gas (hydrogen), a chemical interaction occurs between adsorbed oxygen and hydrogen, releasing electrons back to $CuNiO_2$. In the presence of hydrogen, the above process increases the resistance of $CuNiO_2$ based sensors.

V. SUMMARY AND CONCLUSION

Thin films of copper nickel oxide were formed on to the glass substrates held at room temperature by the method of DC reactive magnetron sputtering the copper nickel alloy ($Cu_{50}Ni_{50}$) target in a mixture of reactive gas of oxygen and sputter gas of argon. The as-deposited films were annealed in air for one hour at temperatures of 200ºC. The chemical composition, crystallographic structure and surface morphology, electrical and optical properties of the as-deposited and annealed films were studied. The as-deposited films were X-ray amorphous. The films annealed at temperature of 200ºC were $CuNiO_2$ with polycrystalline in nature. The annealing of films leads to enhancement in the size of the crystallites due to improvement in the crystallinity. The increase in the electrical resistivity with the increase of annealing temperature was due to filling of oxygen ion vacancies in the films. In conclusion, the $CuNiO_2$ thin films formed by DC reactive magnetron sputtering subsequently annealed in air at 200ºC were polycrystalline in nature with electrical resistivity of 22 Ωcm and optical band gap of 2.06 eV and hydrogen gas sensitivity of 50%.

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