Sensing Behavior of Screen Printed Nanocrystalline Copper Oxide Thick Films in Presence of H_2S Atmosphere

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ABSTRACT

In the present endeavour, CuO nanoparticles were synthesized using sol gel method by sintered at higher temperature 400°C.Nanocrystalline copper oxide (CuO) thick films were deposited on glass substrate by using screen printing technique. Prepared films were characterized by XRD, FTIR, SEM, and EDS for their structural, crystalline size, and morphological properties. The nanoparticles size calculated by Scherer formula for CuO was found to be 19.27nm. The scanning electron microscopy shows prepared material is highly crystalline and showed a homogeneous surface with voids and cavities in various sized nanoparticles.EDS analysis confirms the fundamental elemental composition of copper oxide material. FT Infra-red studies show a characteristic Cu-O stretch for prepared copper oxide. Thick films prepared by screen printing technique were utilized for H_2S sensing. Prepared CuO thick films found to be sensitive towards H_2S gas. The sensitivity of copper oxide films to H_2S was observed as 87% at gas concentration of 100 ppm at room temperature (40^0C). Prepared films were showed good response and recovery time.

Keywords: CuO, Gas sensitivity; Screen printing technique; H_2S; XRD; FTIR; SEM, and EDS.

INTRODUCTION:

Hydrogen sulfide has a very low odor threshold, with its smell being easily perceptible at concentrations well below 1 part per million (ppm) in air. The odor increases as the gas becomes more concentrated, with the strong rotten egg smell recognizable up to 30 ppm. Above this level, the gas is reported to have a sickeningly sweet odor up to around 100 ppm. However, at concentrations above 100 ppm, a person's ability to detect the gas is affected by rapid temporary paralysis of the olfactory nerves in the nose, leading to a loss of the sense of smell. This means that the gas can be present at dangerously high concentrations, with no perceivable odor. Prolonged exposure to lower concentrations can also result in similar effects of olfactory fatigue. This unusual property of hydrogen sulfide makes it extremely dangerous for human being, so it is necessary to control and monitor H_2S gas in the environment [1-5].

Copper oxide (CuO) is also known as cupric oxide. CuO nanomaterials have grown substantially due to its direct band gap, low cost fabrication and good electrochemical properties. CuO is intrinsic p-type semiconductors with relatively small band gap 1.2eV. [6]

Copper oxide nanoparticles have many attractive properties that can be utilized in a different application such as solar cells, as catalytic support materials, as solid-state chemical sensors, solar energy transformation, magnetic storage media, photocatalyst, optoelectronics and potential applications in various fields.

In the present work the synthesis and characterization of copper oxide CuO nanoparticles powder by sol gel method and its sensitivity to H_2S gas at low temperature on prepared thick films were studied.

EXPERIMENTAL DETAILS:

Synthesis of CuO nanoparticles using sol gel method:

CuO nanoparticles have been synthesized by a sol-gel method using cupric acetate Cu (CH_3COO).H_2O as a source of Cu. In a typical experiment; 2.50 gm of cupric acetate was added to 30 ml of methanol and stirred vigorously at 40°C for 2 hr, leading to the formation of cyan powder. The prepared powder was sintered at 400°C using silica crucible annealing time of 2 hours in an ambient temperature to obtain nano crystalline CuO powder. The nanocrystalline CuO powder was further dissolved in 30 ml *m*-cresol and solution was continuously stirred for 22 hr at 60°C temperature and filtered. After that nanoparticles of CuO obtained [6].

RESULTS AND DISCUSSION:

Electrical Characterization:

The electrical characterization of prepared thick film was carried out by using gas sensing system. The series of readings were performed on the CuO sample shown in Fig.1.1 and 1.2.

Fig. 1.1

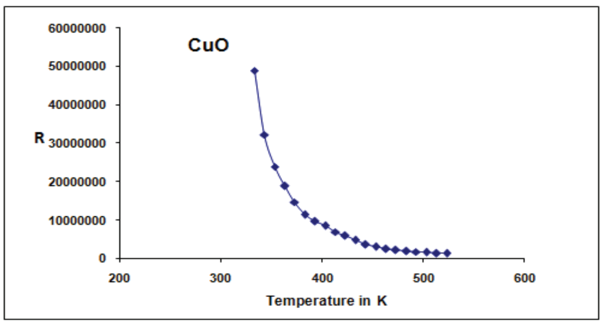
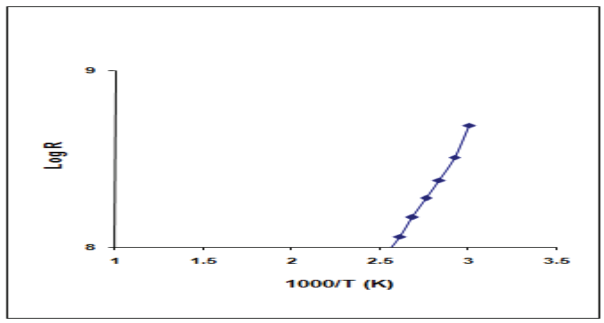


Fig.1.2

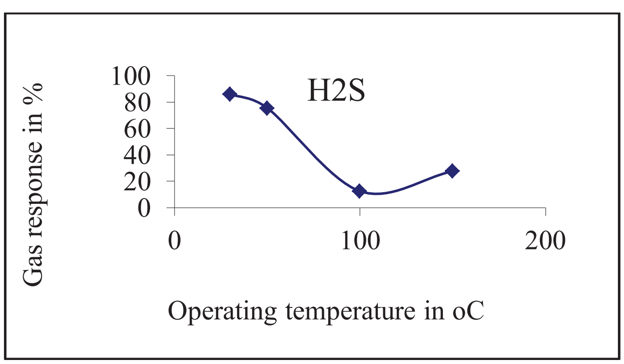


From fig.1.1 and 1.2 confirming semiconducting behavior of prepared nano CuO thick films. The resistance of CuO decreases with increasing temperature it shows NTC.

Gas sensing properties of prepared CuO thick films to H2S:

Fig.2 shows the H_2S gas response was found maximum at room temperature for 100 ppm. The response of CuO thick films to H_2S gas is 87% at room temperature. In present work, every time prior to exposing the CuO film to H_2S, it was allowed to stabilize at an operating temperature for 10 min and the stabilized resistance was taken as Ra. After exposing the film to the H_2S gas, the changed resistance was taken as Rg. H_2S is reducing gas. It reacts with surface oxygen ions of the film. Reduction of film increases the number of free carriers. Therefore resistance of the film increases with reducing gas [11].

Fig.2: Shows H_2S gas sensing response on CuO thick film.

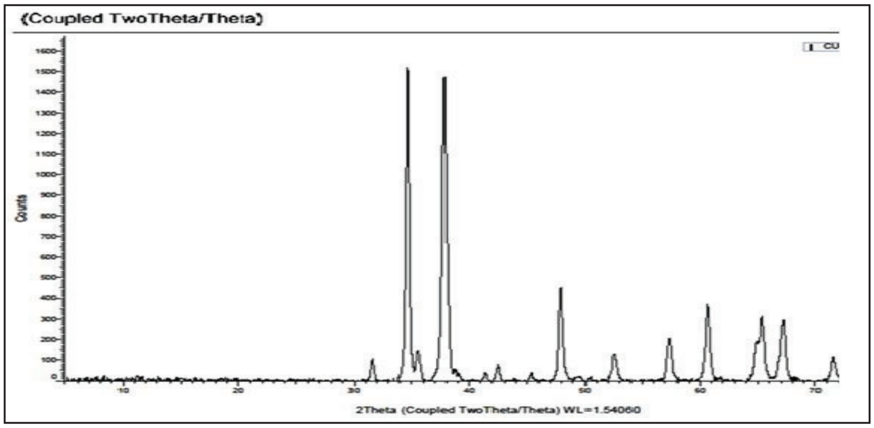


Characterization techniques:

X-Ray Diffraction:

The X-ray diffraction pattern of CuO thick film is shown in figure 3. The XRD analysis confirms the nanoparticles of CuO found to be monoclinic phase. The obtained peaks were comparable to the CuO JCPDS card no: 05-661 data. The sharpness of peaks shows that CuO NPs are highly crystalline. The crystalline size of CuO nanoparticles calculated using FWHM of from most intense XRD peak using Scherrer's formula. Crystalline size for CuO NPs is found to be 19.27 nm [8].

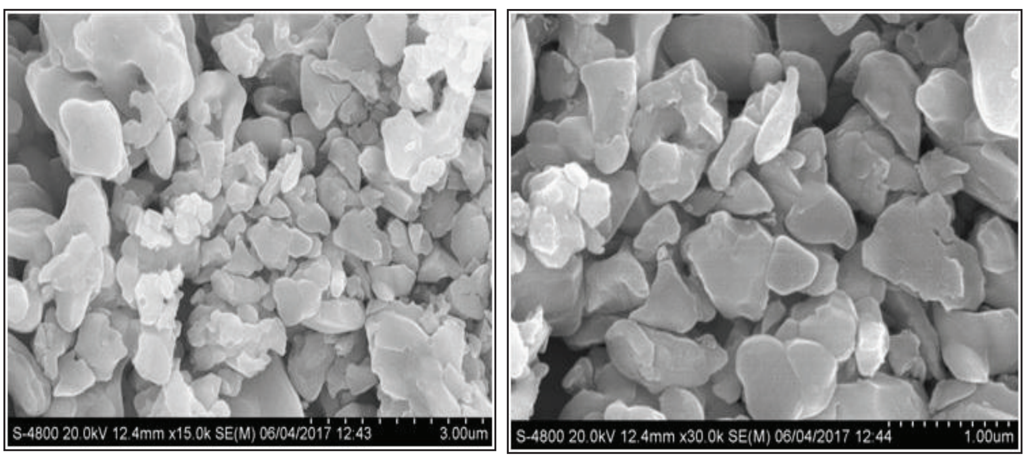
Fig.3 XRD of CuO thick film



Scanning Electron Microscope:

CuO SEM images as depicted in fig.4 showing the nanoparticles with varied nanosized. The SEM images indicating the homogeneous surface of grayish CuO nanoparticles with some small cavities embedded along with nanoparticles. The highly porous surface of CuO thick films is effective for gas sensing phenomenon. [10, 11]

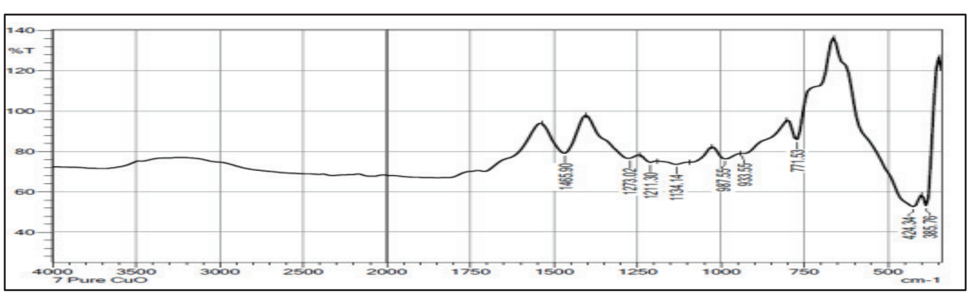
Fig.4: SEM image of prepared CuO Thick Film



FTIR:

The FTIR spectrum of copper oxide thick films as shown in fig.5 indicating the typical copper oxide stretching band observed at 413.24 CM-[1]. FT Infra-red studies show a characteristic Cu-O stretch for prepared copper oxide [8].

Fig. 5: FTIR of CuO Thick film



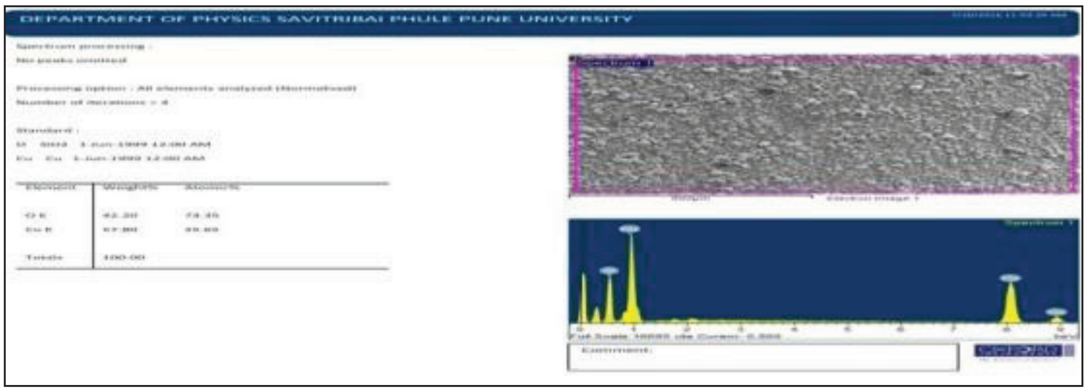
EDS:

Table-1 shows the composition of CuO thick films fired at 450 ^{o}C. EDS is broadly used to determine the elemental composition of MOS which creates element composition images over a much large area together, this information provides information for a variety of materials. The EDAX spectrum shown in fig.6 the presence of only Cu and Oxygen, implying the perfect elemental composition of prepared copper oxide thick films.[11]

Table-1: Composition of the films at 450 ^{0}C firing temperature

Element	Mass %	At. Wt. %
O	42.20	74.35
Cu	57.80	25.65
Total	**100.00**	**100.00**

Fig.6: EDS Spectrum of prepared CuO thick film



CONCLUSIONS:

CuO nanoparticles were successfully synthesized by sol gel method. The nanoparticles size calculated by Scherer formula for CuO was found to be 19.27nm.Thick films of CuO were prepared by screen printing technique. Prepared thick films of copper oxide shows semiconductor behavior. Nanoparticles size and morphological structure confirmed by XRD and SEM, EDS, FTIR studies show a characteristic Cu-O stretch for prepared copper oxide. As regards the gas sensing property of copper oxide films, we can conclude that nano CuO thick films are promising sensors to H_2S at room temperature at low ppm.

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