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HYDROTHERMAL SYNTHESIS OF ZNO NANOPARTICLES AND STUDY THE EFFECT OF DOPING ON ELECTRICAL CONDUCTIVITY OF ZNO THICK FILMS.

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Abstract: This paper covers the synthesis, characterization and electrical behavior of zinc oxide nanoparticles. Hydrothermal technique was used for synthesis of ZnO nanoparticles. The particle size of synthesized ZnO calculated theoretically from XRD and confirmed By TEM. Morphology of synthesized material studied from SEM. Electrical conductivity of the thick films were studied at different temperature and compared the electrical conductivity of ZnO thick films by using different concentrations of dopant. Briefly discusses the electrical conductivity of doped and undoped zinc oxide thick films and influence of doping on electrical conductivity were discussed. Electrical conductivity measurements at room temperature show semiconductor nature of films. The electrical conductivity of Cu-doped ZnO thick film was lower than that of the undoped ZnO thick film. Because of doped Cu builds the potential barrier in the grain boundary of ZnO and made the grain boundary region more resistive.

Keywords: Hydrothermal Synthesis; Nanosize ZnO; thick films; Electrical Conductivity.

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INTRODUCTION

Zinc oxide efficiently utilized for various technological applications such as antireflection coatings, transparent electrodes in solar cells [1,2], piezoelectric devices [3], varistors [4], surface acoustic wave devices [5], electro and photo luminescent devices [6], gas sensors [7,8], and others. Now zinc oxide has attracted worldwide research interest because it is considered a promising material for gas sensors in electronic noses [9,10]. Although ZnO is one of the earliest discovered semiconducting oxide gas sensing materials and there are many reports concerning the sensitivity properties of ZnO, most of these works has been done on powder samples usually pressed in pellets and sintered at high temperatures (900–1300 °C) [11,12]. In recent years, there appeared many publication on ZnO nanorod or thin film using various synthesis techniques including molecular beam epitaxy [13–17], chemical vapor deposition [18,19], sputtering [20,21], thermal evaporation [22,23], and reactive vapor deposition [24]. The hydrothermal technique for synthesis of nanoparticles is low temperature and environmental friendly process.

2. Experimental details

All the chemicals were used in the present work of AR grade without further purification. 2g $Zn(AC)_2$ was dissolved in 110 ml de-ionized (DI) water by 10 min stirring. Subsequently, 10 ml 2 M NaOH aqueous solution was introduced into the above aqueous solution result in a pH of 12. The solutions were then transferred into Teflon lined stainless steel autoclaves, sealed and maintained at 160 ^{0}C for 20 h, the resultant white solid product were centrifugalize, washed with DI water and ethanol to remove the ions possibly remaining in the final products, dried at 60 ^{0}C in air. The dried powder was use for preparation of thixotropic paste.

2.1 thick films preparation

The prepared thixotropic paste was used to preparation of thick films of ZnO on glass substrate by using Screen printing method. Some of thick ZnO films were doped by dipping them into a 0.01 M aqueous solution of cupper chloride ($CuCl_2$) for 5 min., 10 min and 15 min intervals of time. A fresh solution was used for each dipping. These films were dried in air for half an hour. These films are termed as CuO modified ZnO thick films.

2.2 measurement of electrical conductivity

Electrical conductivity of all the thick films was measured by conductivity measurement setup.

3. RESULTS AND DISCCUSTION

3.1 x-ray diffraction pattern

Obtained X-ray pattern of the synthesized ZnO was matched with the reported JCPDS data (card no. 05-0664). The crystal structure of the samples were determine. The zinc oxide was found to be of wuartzite structure with hexagonal symmetry .

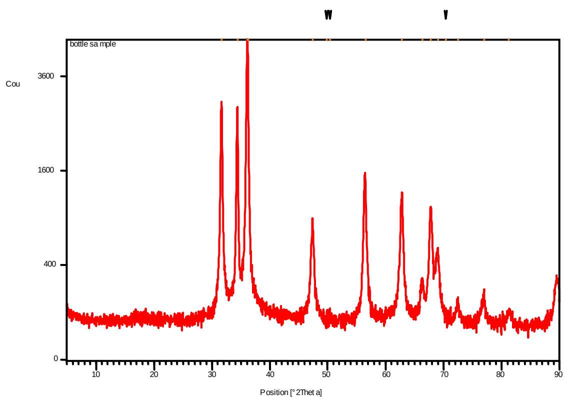

Fig.1. X-ray diffraction pattern of ZnO nanoparticles

The diffraction patterns of nano- ZnO are shown in fig.1 The particle size were determined from the line broadening of the diffraction lines corrected for instrumental broadening, using scherre equation [1]. The average particle size of nano ZnO were ~ 45 nm.

3.2 scanning electron microscope (SEM)

The SEM image of Synthesized ZnO nanoparticles as shown in fig.2

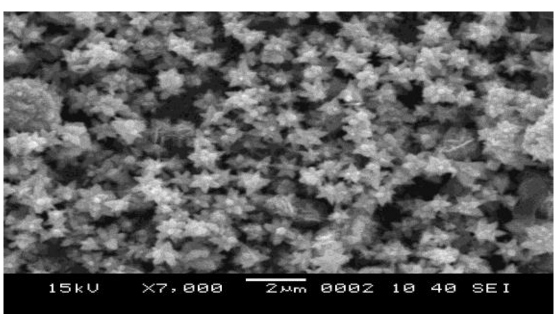

Fig. 2. SEM image of nano ZnO prepared by hydrothermal process.

Surface morphology and structure were observed by SEM. Form the picture it is observed that the ZnO particles are aggregate star like structure.

3.3 transmission electron microscope (TEM)

TEM image of Synthesized ZnO nanoparticles as shown in fig.3

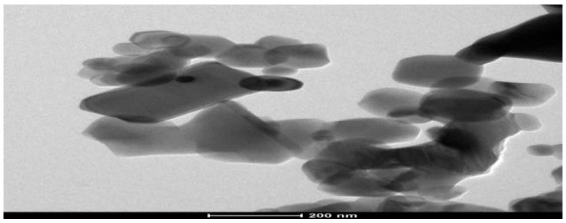

Fig.3. TEM images of nano ZnO prepared by hydrothermal process.

It has been seen that the Synthesized ZnO material by hydrothermal process has grain size in nanometer range which strongly agreed with the theoretically calculated grain size by using Scherrer formula. A small grain size which results in an increase in porosity

3.4 measurement of a.c. electrical conductivity

a.c. electrical conductivity measured by using Agilent 4284A LCR meter. Fig. 4(a), (b), (c), (d) shows variation a.c. electrical conductivity with frequency of ZnO, CuO modified ZnO (5 min), CuO modified ZnO (10 min) and CuO modified ZnO (15 min) thick films respectively.

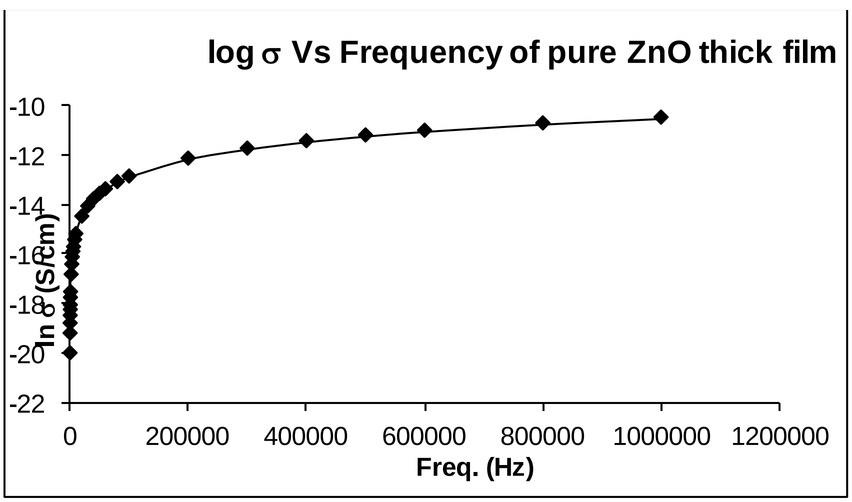

Fig .4.(a) Variation of lnσ with frequency of ZnO thick film.

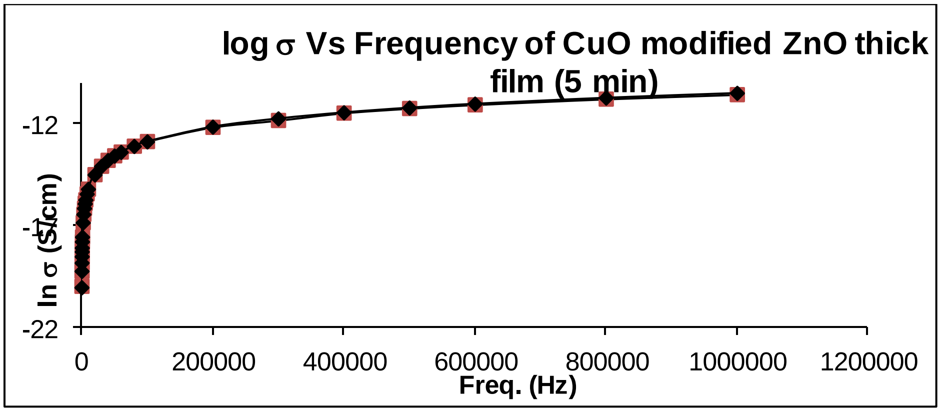

Fig. 4.(b) Variation of lnσ with freq. of CuO modified ZnO (5 min) thick film.

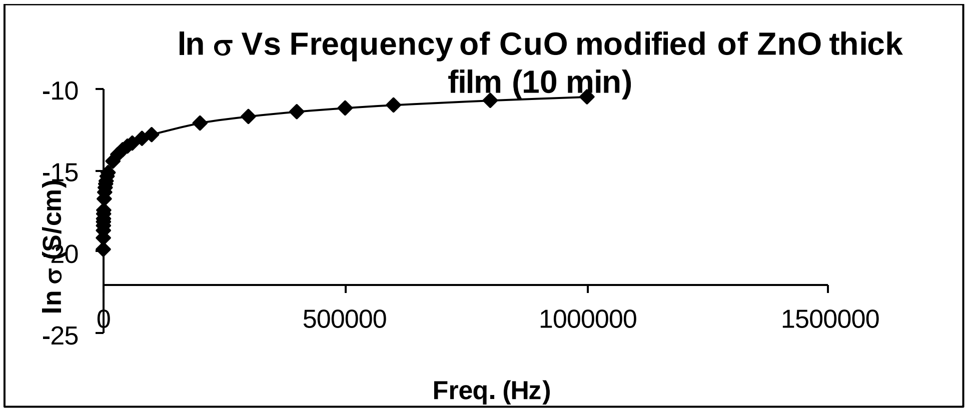

Fig .4. (c) Variation of lnσ with frequency of CuO modified ZnO (10 min) thick film.

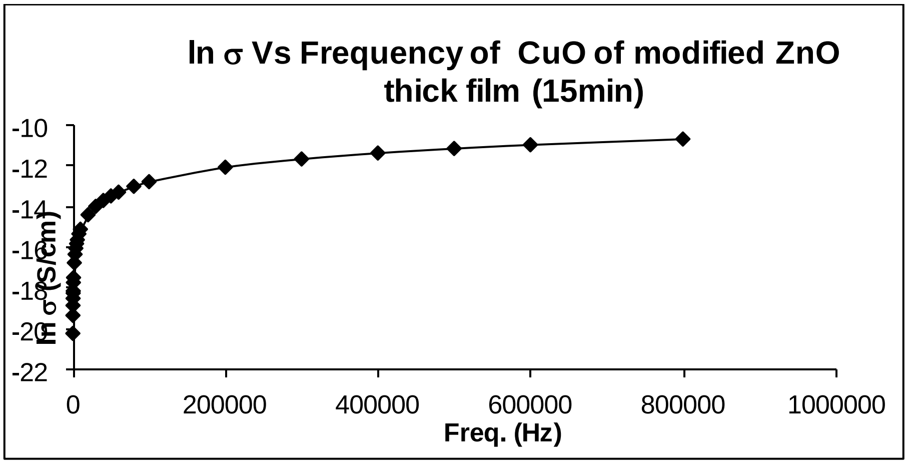

Fig.4. (d) Variation of lnσ with frequency of CuO modified ZnO (15 min) thick film.

3.5. I-V characteristics of film

I-V characteristics of pure and CuO modified ZnO were studied at room temperature. Fig.5. represents I-V characteristics of ZnO, CuO modified ZnO (5 min), CuO modified ZnO (10 min) and CuO modified ZnO (15 min). Variation of current with applied voltage is found to be linear in nature for all thick films. The linear nature I-V characteristics of all thick films indicating the ohmic nature of silver contacts. [1]

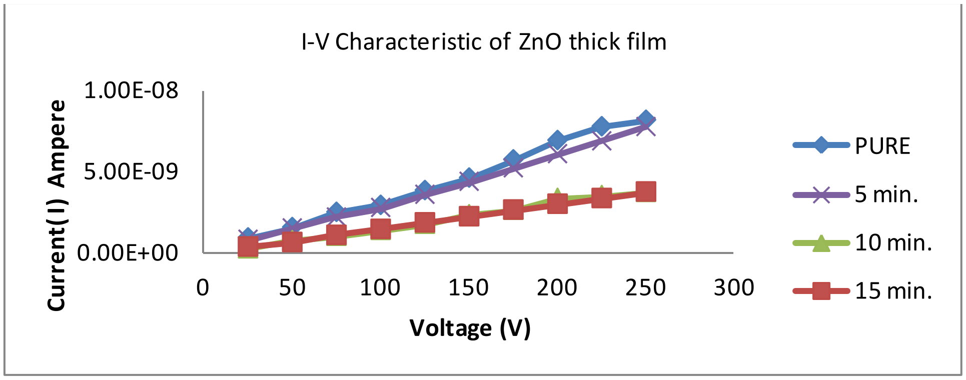

Fig.5. I-V Characteristics of ZnO & CuO modified thick films.

3.6 variation of conductivity with temp.

Fig.6 depicts the variation of log conductivity with the temperature of ZnO, CuO modified ZnO (5 min), CuO modified ZnO (10 min) and CuO modified ZnO (15 min) thick films.

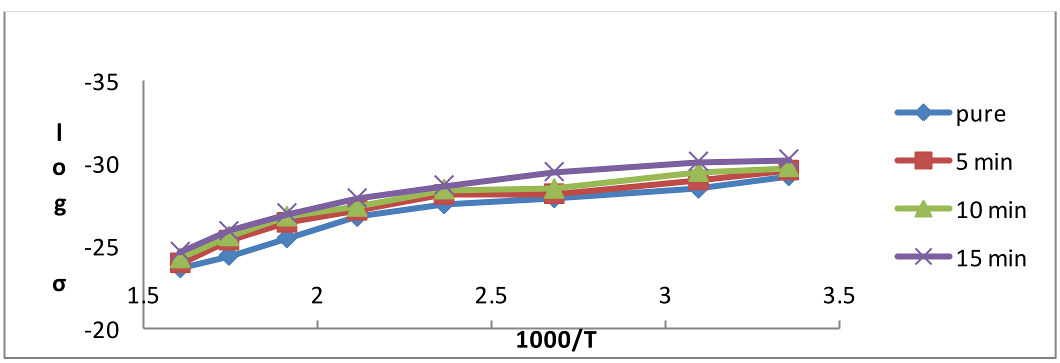

Fig.6. logσ with the temperature of ZnO, CuO modified ZnO 5 min., 10 min. and 15 min. thick films.

The plot between ln σ Vs 1000/T has straight line nature, it indicated that the conductivity varies exponentially with temperature. The semiconducting nature of ZnO is observed form the measurement of conductivity with temperature. Increasing in conductivity with temperature of

ZnO may be due to large oxygen deficiency which could adsorb oxygen species at higher temperature. The adsorption phenomena of the CuO modified ZnO thick film surface would be different from the pure ZnO thick films surface. The CuO misfits on the surface are the places where the oxygen species adsorb. The CuO misfits distributed evenly on the surface would have made it possible to absorb the oxygen ions even at low temperature. Form fig.6 it is clear that conductivity of pure and CuO modified ZnO films increases with an increase in temperature, indicating a positive temperature coefficient of conductance. This behavior confirmed the semiconducting nature of the pure and modified ZnO.

It is observed from fig.6 that the electrical conductivity of the pure ZnO films was larger than modified ZnO films in ambient air. It may be due to the intergranular potential barrier[1] Pure ZnO has only one kind of grains arranged uniformly, where in the case of CuO modified films the grains are of different natures such as CuO and ZnO. The modification causes the formation of heterogeneous intergrain boundaries of the CuO-ZnO. Thus increased barrier heights of the intergranular regions of activated ZnO may be responsible to decrease the conductivity.

The a.c. conductivity of ZnO and CuO modified thick films measured in the frequency range of 100Hz to 1MHz at room temperature. The experimental data reveal that ac conductivity increases with frequency. Variation of a.c. conductivity with frequency can be explained with the help of barrier hopping mode [3].

CONCLUSION

This paper, reported the experimental result about synthesis of nanostructured ZnO material by hydrothermal method and electrical conductivity of ZnO nanostructured doped and undoped thick film. On the basis of experimental result, the conclusions are as follows:

(i) Nanostructured ZnO particles were successfully synthesized. The particles size of synthesized ZnO is in nano order. The powder processing has an important influence on the sizes of the crystallites.

(ii) The homogeneity of the materials confirmed by XRD measurement.

(iii) The electrical conductivity of Cu-doped ZnO thick film was lower than that of the undoped ZnO thick film Because Cu doping led to the formation of acceptor levels in ZnO. The doping of Cu build up the potential barrier in the grain boundary of ZnO and made the grain boundary region more resistive.

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