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Hydrothermal synthesis and characterization of copper oxide flower-like nanostructures

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ABSTRACT

This paper describes the synthesis of Copper oxide nanostructures by a simple cost effective hydrothermal process. The structural analysis reveals that CuO exhibits a monoclinic crystal structure. A variety of other techniques like UV-Vis spectroscopy, Fourier Transform Infrared spectroscopy, Scanning Electron microscopy and dielectric analysis were done to study the optical, morphological and electrical characteristics of the nanomaterial.

Introduction

Copper Oxide is a p-type semiconductor oxide that is being widely studied due to its interesting properties and applications. The possibility for controllable tuning of its chemical and physical properties enables this narrow band gap (1.2eV) metal oxide to fulfill its full potential in different applications. Interdisciplinary research on a wide range of applications such as sensors [1], catalysis [2, 3], solar energy transformers [4-7], lithium ion batteries [8, 9] and photoconductive compounds [10-12] makes it a much sought after oxide.

CuO has a rich genre of morphologies that have been obtained such as nanorods[13], nanowires[14,15], nanoplatelets[16,17], flower-like[18], dendrites[19], cubes [20-22], spherical, star-like, octahedral [23] and so on. In addition, various techniques are employed to synthesize nanostructures, such as, hydrothermal [24-26], solvothermal [27], Electrodeposition[28-31], simple solution[32,33], microwave irradiation [34] and wet chemical reduction[20,22,35]

Herein, we report the synthesis of copper oxide by the hydrothermal method. The prepared sample was characterized structurally, optically and electrically and the results obtained were reported and discussed in the light of literature.

Experimental

Hydrothermal method is a simple and convenient method to produce CuO nanostructures. The chemicals Copper nitrate, Ammonia, PEG and NaOH (Merck Chemical Company) were used as precursors. Throughout the synthesis distilled water was used for aqueous solution preparation. First a light blue solution of copper nitrate was prepared to which ammonia was added under continuous stirring. Next PEG was slowly added to the above solution and stirred for 5 minutes. Finally NaOH was added and stirred vigorously. The obtained solution was transferred into a Teflon lined autoclave and hydrothermally treated at 110°C for 30 hours. The resulting precipitate was collected and washed with distilled water and ethanol and vacuum dried at 60°C for 6 hours.

X-ray powder diffraction was obtained using Bruker D8 Advance X-Ray diffractometer with monochromated CuKα radiation (λ=1.5406 Å). Optical absorption measurement was done at room temperature using a Cary 5E UV-Vis-NIR spectrophotometer in the wavelength range of 200 to 1200 nm. The FTIR analysis was carried out using BRUKER RFS 27: Stand alone FT-Raman Spectrometer. The morphology of the particle was studied by using the Quanta 200 FEG Scanning electron microscope (SEM).

Results and Discussion

The XRD patterns obtained for the CuO particles are shown in figure 1. Comparison with the JCPDS data [89-5899] reveals a fairly well matched monoclinic structure. The lattice parameters were found to be a= 4.638 Å; b=3.435 Å; c= 5.129 Å. The broadening of the peaks observed in the XRD pattern indicates the nanostructure. The average size of the particle observed is found to be between 13.92 nm and 22.08 nm.

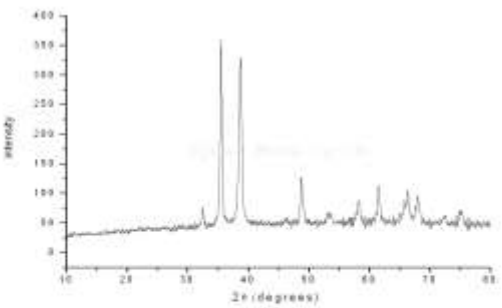

Figure 1 : XRD pattern of the as- synthesized copper oxide particles

The optical absorbance spectrum of Copper oxide was measured between 200 – 1200 nm. It was noticed that the sample is transparent above 400 nm i.e. full visible region. The absorption peak was observed at 962.50nm the band gap thereby calculated was found to be 1.29eV

The FTIR analysis is done to provide information on the molecular structure and chemical bonding of the sample. The

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spectrum was recorded in the wavenumber range 400 – 4000 cm^{-1}.In the spectrum , the absorption band in the region 480 -600 cm^{-1} is attributed to Cu(II)O. It is reported that CuO exhibits 3 peaks around 588, 534, 480 cm^{-1} due to Cu(II)-O vibrations. The peak around 1395 cm-1 can be assigned to the CuO nanoparticles exhibiting a Cu^{2+}-O^{2-} stretch peak.

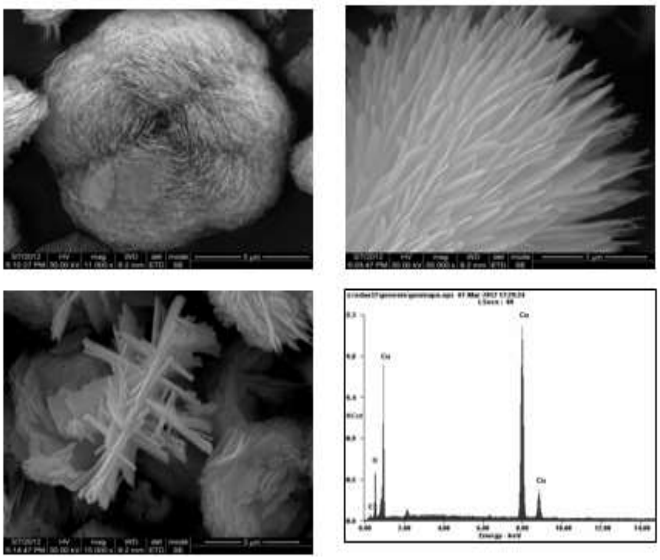
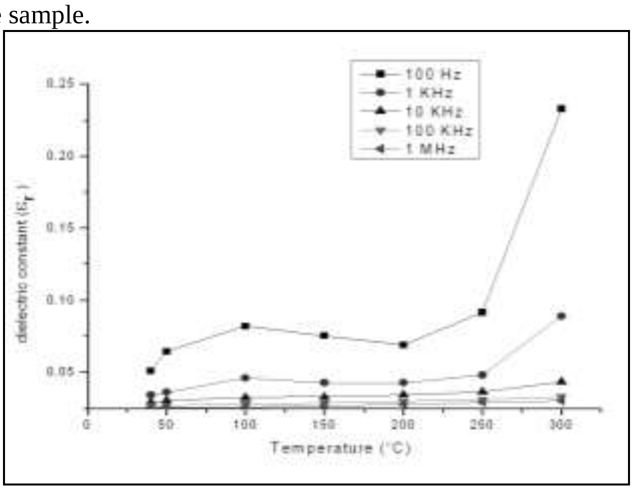
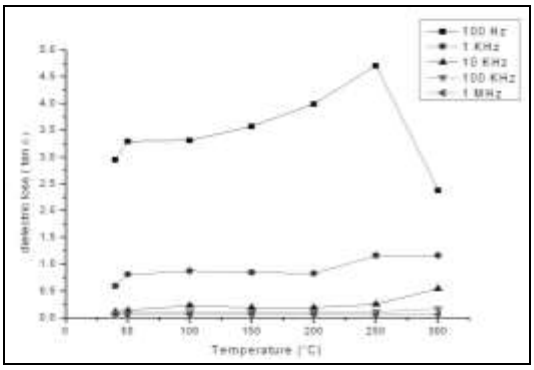


Figure 2 : SEM micrograph of CuO flower nanostructures and the EDS report of the sample

The SEM image recorded for CuO (shown in figure 2) indicates nearly uniform size and flower-like shapes. From the EDS analysis we obtained the constituent elements present and the atomic percentage determined Cu - 68.14 % and O - 18.12%

The dielectric constant (ε_r), loss (tan δ) and AC conductivity measurements were carried out on the sample using an HIOKI 3532 Hitester LCR meter for various temperatures. All the electrical parameters ie ε_r, tan δ and σ_{ac} are seen to increase with the increase in temperature. The dielectric constant was determined using the relation $\varepsilon_r = C_pT/ \varepsilon_o A$, C_p - capacitance of the sample measured directly by the LCR meter, ε_o - permittivity of free space (8.85 x 10^{-12} $C^2 N^{-1} m^{-2}$).The dielectric constant is due to space charge, ionic, dipole and electronic polarization. The increase in ε_r is mainly attributed to space charge and dipole polarization which is strongly temperature dependent. The AC electrical conductivity was determined using the relation $\sigma_{ac} = \omega\, \varepsilon_o\, \varepsilon_r \tan\delta$ ($\omega = 2\pi f$, f is the frequency). With the high AC resistance it can be mentioned that the space charge polarization plays an important role in the electrical property of the sample.





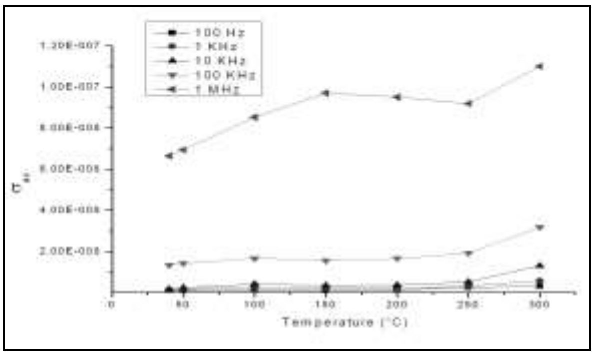


Figure 3 : Electrical Analysis of the sample a) dielectric constant vs temperature;

Conclusion

We have synthesized copper oxide nanostructures with flower – like morphology using a simple hydrothermal process. The XRD and SEM results confirm the nanocrystalline nature of the as-synthesized products. The UV-Vis and FTIR spectra of the particles have also been investigated. The electrical analysis of the sample indicates that space polarization plays an important role in determining the electrical property of the sample.

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