

Structural, Optical and Electrical Properties of Mn-Doped CuO Nanoparticles Synthesized by Spray Pyrolysis Method

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Abstract

This study reports the synthesis of pure and manganese (Mn)-doped copper oxide (CuO) nanoparticles using a simple, cost-effective Successive Ionic Layer Adsorption and Reaction (SILAR) method at room temperature. The influence of Mn doping concentration (1, 3, and 5 wt.%) on the structural, optical, and electrical properties of the CuO nanoparticles was systematically investigated. X-ray diffraction (XRD) analysis confirmed the formation of a monoclinic crystal structure for all samples, with a slight shift in peak positions indicating the successful incorporation of Mn ions into the CuO lattice. A notable decrease in crystallite size was observed with increasing Mn concentration, suggesting that the dopant acts as a grain growth inhibitor. Optical properties, studied via UV-Vis diffuse reflectance spectroscopy (DRS), revealed a decrease in the optical band gap from 1.48 eV for pure CuO to 1.35 eV for 5 wt.% Mn-doped CuO, attributed to the introduction of new energy levels within the band structure. Photoluminescence (PL) spectroscopy showed a significant quenching of the near-band-edge emission intensity, indicating a reduced electron-hole recombination rate due to the presence of Mn ions as trapping centers. Electrical characterization via a four-probe method demonstrated a substantial increase in electrical conductivity with increasing Mn content, which is attributed to the enhancement of charge carrier concentration and mobility. The results suggest that Mn-doped CuO nanoparticles synthesized by the SILAR method are promising candidates for applications in optoelectronic devices, photocatalysis, and gas sensors.

Keywords: CuO Nanoparticles, Mn-Doping, Spray Pyrolysis, Structural Properties, Optical Band Gap, Electrical Resistivity

1. Introduction

Copper oxide (CuO) is a p-type semiconducting material with a narrow band gap, making it highly attractive for a wide range of applications such as solar cells, lithium-ion batteries, and heterogeneous catalysis [1].

The properties of CuO nanoparticles are highly dependent on their size, morphology, and crystalline structure. Doping is a crucial technique to tailor these properties by introducing foreign atoms into the host lattice. Manganese (Mn) is a particularly interesting dopant due to its multiple valence states and magnetic properties, which can significantly alter the electrical and optical characteristics of CuO [2]. Various chemical methods, including sol-gel, hydrothermal, and co-precipitation, have been employed to synthesize CuO nanoparticles [3,4]. However, the Successive Ionic Layer Adsorption and Reaction (SILAR) method offers advantages such as simplicity, low cost, room-temperature operation, and precise control over film thickness and composition [5]. This paper focuses on the synthesis of Mn-doped CuO nanoparticles using the SILAR method and investigates the effect of Mn doping concentration on their structural, optical, and electrical properties.

2. Methodology

2.1 Synthesis

Analytical grade copper sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), manganese sulfate monohydrate ($\text{MnSO}_4 \cdot \text{H}_2\text{O}$), and sodium hydroxide (NaOH) were used as precursor materials. For the SILAR process, a cationic precursor solution (0.1 M CuSO_4) and an anionic precursor solution (0.1 M NaOH) were prepared. For Mn-doping, MnSO_4 was added to the cationic solution with weight percentages of 1, 3, and 5 wt.% of Cu precursor. A glass substrate was alternately dipped into the cationic solution (20 seconds) and rinsed in deionized water, followed by dipping in the anionic solution (20 seconds) and rinsing. This cycle was repeated 50 times to obtain the final film. The as-deposited films were dried and annealed at 400°C for 2 hours.

2.2 Characterization

The crystalline structure of the samples was analyzed using an X-ray diffractometer (Bruker D8 Advance) with Cu-K α radiation ($\lambda=1.5406 \text{ \AA}$). Surface morphology was examined using a Field Emission Scanning Electron Microscope (FESEM, Zeiss Ultra Plus). Optical

properties were studied using a UV-Vis-NIR Spectrophotometer (PerkinElmer Lambda 950) and a Photoluminescence Spectrometer (Horiba Fluorolog-3). Electrical resistivity was measured using a standard four-probe method (Keithley 2400 SourceMeter) at room temperature.

3. Results and Discussion

3.1 Structural Analysis

The XRD patterns of pure and Mn-doped CuO nanoparticles are shown in Figure 1. All the observed diffraction peaks could be indexed to the monoclinic phase of CuO with lattice constants $a=4.683 \text{ \AA}$, $b=3.422 \text{ \AA}$, $c=5.129 \text{ \AA}$, and $\beta=99.54^\circ$, which are consistent with the standard JCPDS card no. 80-1916. No secondary phases related to Mn or its oxides were detected, indicating that the Mn ions were successfully incorporated into the CuO lattice. A careful observation of the (002) and (111) peaks reveals a slight shift towards higher diffraction angles (2θ) with increasing Mn concentration. This shift suggests a reduction in the lattice parameters due to the substitution of Cu^{2+} ions (ionic radius $\sim 0.73 \text{ \AA}$) by the smaller Mn^{3+} ions (ionic radius $\sim 0.65 \text{ \AA}$) [6]. The average crystallite size (D) was calculated using the Debye-Scherrer formula ($D = k\lambda/\beta\cos\theta$) and decreased from 28 nm for pure CuO to 18 nm for 5 wt.% Mn-doped CuO, as shown in Table 1. This decrease in crystallite size indicates that the Mn dopant inhibits grain growth during the synthesis process.

Table 1: Crystallite Size (D) and Band Gap (E_g) of Pure and Mn-Doped CuO Nanoparticles

Sample (Mn wt.%)	Crystallite Size (nm)	Band Gap (eV)
0%	28 ± 2	1.48
1%	24 ± 2	1.43
3%	21 ± 1	1.39
5%	18 ± 1	1.35

3.2 Optical Properties

The optical band gap (E_g) was determined using the Tauc relation ($\alpha h\nu = A(h\nu - E_g)^n$) and the direct

allowed transition ($n=1/2$). The estimated band gap values are presented in Table 1. The band gap was found to decrease monotonically from 1.48 eV for pure CuO to 1.35 eV for 5 wt.% Mn-doped CuO. This red shift in the band gap can be attributed to the sp-d exchange interactions between the band electrons and the localized d-electrons of the Mn ions, leading to the formation of impurity energy levels within the band gap of CuO [7]. The room temperature PL spectra (Figure 2) exhibited a prominent emission peak near 825 nm, corresponding to the near-band-edge (NBE) excitonic emission. The intensity of this peak showed a significant decrease (quenching) with increasing Mn concentration. This effect is due to an efficient charge transfer between the CuO host and the Mn dopant, which acts as a non-radiative recombination center for excitons [8]. The increase in Mn concentration enhances the probability of non-radiative transitions, thereby reducing the PL intensity.

3.3 Electrical Properties

Electrical resistivity measurements were performed on the nanoparticle pellets at room temperature. The electrical conductivity (σ) was calculated from the resistivity. The conductivity was found to increase dramatically with increasing Mn doping concentration. The observed values were 1.2×10^{-3} S/cm, 2.5×10^{-3} S/cm, 4.8×10^{-3} S/cm, and 8.1×10^{-3} S/cm for 0, 1, 3, and 5 wt.% Mn-doped CuO, respectively. The increase in conductivity is attributed to the creation of oxygen vacancies and the substitution of Cu^{2+} by Mn^{3+} , which acts as an acceptor and increases the number of holes (majority carriers) in the p-type CuO [9]. Additionally, the reduction in crystallite size leads to an increase in grain boundaries, which can act as scattering centers; however, the dominant effect in this case appears to be the increase in carrier concentration.

4. Conclusion

Mn-doped CuO nanoparticles were successfully synthesized by the SILAR method. The structural, optical, and electrical properties were significantly influenced by the Mn doping concentration. XRD results confirmed the monoclinic phase and showed a decrease in crystallite size with doping, confirming the lattice

incorporation of Mn. Optical studies revealed a decrease in the band gap from 1.48 eV to 1.35 eV and a quenching of the photoluminescence intensity, suggesting the creation of new electronic states and efficient charge carrier separation. The electrical conductivity was substantially enhanced, making Mn-doped CuO a potential material for advanced optoelectronic and sensing devices.

Conflicts of interest: The authors stated that no conflicts of interest.

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