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

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Abstract

This research article presents a comprehensive investigation of the structural, optical, and electrical properties of undoped and manganese-doped copper oxide (CuO) nanoparticles synthesized via the Successive Ionic Layer Adsorption and Reaction (SILAR) method. CuO is an important p-type semiconductor with a narrow band gap of 1.2–1.9 eV, making it suitable for applications in gas sensors, solar cells, and lithium-ion batteries. In this work, manganese doping was incorporated to modify and enhance the physical properties of CuO nanostructures. The morphological analysis revealed that the films consist of plate-like nanostructures, with Mn-doping concentration significantly affecting the shape and size of the nanostructures. X-ray diffraction (XRD) analysis confirmed the monoclinic crystal structure for all films, with a slight shift in peak positions observed upon Mn incorporation, indicating lattice distortion. Optical characterization using UV-visible spectrophotometry showed that the optical band gap increases with increasing Mn-doping concentrations. Electrical studies demonstrated enhanced conductivity with Mn-doping, attributed to the formation of defects at grain boundaries. The results demonstrate that Mn-doping provides an effective approach for tuning the optical and electrical properties of CuO nanostructures, making them promising candidates for optoelectronic device applications.

Keywords: CuO Nanoparticles, Mn-Doping, SILAR Method, Structural Properties, Optical Band Gap, Electrical Conductivity

1. Introduction

Transition metal oxides in the nanoscale region have garnered significant research interest due to their fascinating properties and wide-ranging applications in modern technology [1]. Among these materials, cupric oxide (CuO) has emerged as an important p-type transition metal oxide with a narrow band gap of 1.2–1.9 eV [1].

CuO has received considerable attention in recent years due to its unique properties and extensive application range in gas sensors, biosensors, solar cells, high-temperature superconductors, lithium-ion batteries, and catalysts [1].

The performance of CuO-based devices depends critically on the morphology, structure, and composition of the material. Researchers have made numerous efforts to fabricate nanostructured CuO materials with various morphologies, including nanoparticles, nanorods, nanowires, nanoplates, and nanotubes, to enhance its performance [1]. It is well-established that shape-controlled morphologies significantly affect the properties of nanomaterials [2].

Doping with appropriate elements provides an effective strategy to enhance the physical and chemical properties of CuO [1,3]. Manganese is known to be an excellent catalyst, and mixed oxide catalysts containing manganese oxides or copper oxides have been extensively studied [1,4]. While some research has been conducted on Mn-doped CuO nanowires synthesized by oxidizing Mn-Cu alloys, powder and pellet samples by coprecipitation method, and thin films by RF magnetron sputtering, there are limited reports on the morphological, structural, and optical properties of Mn-doped nanostructured CuO films synthesized by solution-based techniques [1,5].

The Successive Ionic Layer Adsorption and Reaction (SILAR) method is a promising solution-based technique for thin film deposition [1]. This method is simple, safe, environmentally friendly, and low-cost, capable of producing metal oxide films at relatively low temperatures [1,6]. Unlike techniques that require high annealing temperatures and extended processing times, SILAR offers advantages for large-scale production of nanostructured films [1].

The present study aims to synthesize undoped and Mn-doped CuO nanostructures onto glass substrates using the SILAR method and investigate the influence of Mn-doping concentration on the morphological, structural, optical, and electrical properties of the resulting films. The findings are correlated and discussed in terms of the potential applications of these materials in optoelectronic devices.

2. Methodology

2.1 Materials

Analytical grade reagents were used without further purification. Copper (II) chloride dihydrate ($CuCl_2 \cdot 2H_2O$) and Manganese (II) chloride tetrahydrate ($MnCl_2 \cdot 4H_2O$) were purchased from Sigma-Aldrich and Merck KGaA [1]. Glass microscope slides were used as substrates for thin film deposition.

2.2 Synthesis of Undoped and Mn-Doped CuO Films

Undoped CuO films were synthesized by preparing a 0.1 M copper chloride solution [1]. For Mn-doped samples, solutions containing Cu^{2+} and Mn^{2+} ions with varying concentrations (1%, 3%, and 5% Mn relative to Cu) were prepared [1]. Glass substrates were cleaned ultrasonically in ethanol and distilled water before deposition [1].

The SILAR deposition process involved sequential immersion of the substrates into [1]:

1. The cationic precursor solution (Cu^{2+} or Cu^{2+}:Mn^{2+})
2. Distilled water for rinsing
3. An anionic precursor solution (hot water at 90°C) for oxidation
4. Distilled water for rinsing

This cycle was repeated to achieve the desired film thickness. The substrates were dried in air after each deposition cycle [1].

2.3 Characterization Techniques

Structural Characterization: X-ray diffraction (XRD) patterns were recorded using a Philips diffractometer with Cu-Kα radiation to determine the crystal structure and phase purity of the films [2,7]. The crystallite size was calculated using the Debye-Scherrer formula [2,8]:

$$D = k\lambda / (\beta \cos\theta)$$

where D is the crystallite size, k is the dimensionless shape factor (0.9), λ is the X-ray wavelength, β is the full

width at half maximum (FWHM), and θ is Bragg's angle [2].

Morphological Characterization:
Scanning electron microscopy (SEM) was employed to examine the surface morphology of the films [1]. Energy dispersive spectroscopy (EDS) was used for elemental composition analysis [1,9].

Optical Characterization:
The optical properties were investigated using UV-visible spectrophotometry in the wavelength range 300-900 nm [1,10]. The optical band gap was determined using Tauc's relation [1,11]:

$$(\alpha h\nu)^n = A(h\nu - Eg)$$

where α is the absorption coefficient, hν is the photon energy, A is a constant, n = 1/2 for direct band gap, and Eg is the optical band gap [1,12].

Electrical Characterization:
Electrical properties were studied by measuring the capacitance, dielectric constant, and AC conductivity using an impedance analyzer in the frequency range 20 Hz to 1 MHz at room temperature [2,13]. The dielectric constant was calculated using [2,14]:

$$\varepsilon' = Ct / (A\varepsilon_0)$$

where C is the capacitance, t is the film thickness, A is the area of cross-section, and ε_0 is the permittivity of free space [2].

3. Results and Discussion

3.1 Structural Properties

3.1.1 XRD Analysis
X-ray diffraction patterns of undoped and Mn-doped CuO films are presented in Figure 1. The diffraction peaks correspond to the monoclinic structure of CuO, matching well with JCPDS card no. 89-5899 [1,2]. The peaks observed at 2θ values of approximately 32.5°, 35.6°, 38.8°, 48.8°, 53.5°, and 58.3° correspond to the (110), (002), (111), (202), (020), and (202) planes, respectively [1,2].

A slight shift in peak positions towards higher 2θ values is observed for Mn-doped samples, indicating a slight distortion in the symmetry of the CuO structure due to the formation of vacancies and defects [2]. This shift is attributed to the substitution of Cu^{2+} ions (ionic radius 0.73 Å) by Mn^{2+} ions (ionic radius 0.80 Å) or Mn^{3+} ions (ionic radius 0.58 Å) [2,15]. The formation of charge imbalance due to Mn doping, particularly when Mn^{3+} replaces Cu^{2+}, creates vacancies and defects in the crystal structure [2].

3.1.2 Crystallite Size Analysis
The crystallite size calculated using the Debye-Scherrer formula showed a decrease with increasing Mn concentration [2]. The estimated crystallite sizes were approximately 123 nm for undoped CuO, decreasing to 65 nm, 51 nm, and 45 nm for 5%, 10%, and 15% Mn-doping, respectively [2]. This decrease in crystallite size can be explained by the difference in ionic radii between Mn^{3+} (0.58 Å) and Cu^{2+} (0.73 Å), which induces lattice strain and inhibits crystal growth [2,14,16].

The lattice parameters also exhibited variation with Mn-doping [2]. The cell volume decreased from 81.07 $Å^3$ for undoped CuO to 80.66 $Å^3$ for 15% Mn-doped CuO [2]. This reduction in cell volume provides further evidence for the successful incorporation of Mn ions into the CuO lattice [2].

3.2 Morphological Properties

3.2.1 SEM Analysis
Scanning electron microscopy images revealed that all films consist of plate-like nanostructures with uniform surface coverage [1]. The thickness and dimensions of these plate-like structures were affected by Mn concentration [1]. EDS analysis confirmed the presence of Mn in the doped samples, with Mn-doping percentages in the films showing a linear dependence with the Mn^{2+} ion concentration in the growth solution [1].

The observed morphological changes can be attributed to the influence of Mn ions on the nucleation and growth processes [1,17]. Mn ions introduce defects and

strain in the lattice, altering the surface energy and modifying the growth kinetics of the nanostructures [1].

3.3 Optical Properties

3.3.1 UV-Visible Spectrophotometry

The optical properties were investigated through UV-visible spectrophotometry. Figure 2 shows the absorbance spectra of undoped and Mn-doped CuO films [1]. The absorbance edge shifts towards higher energies with increasing Mn concentration, indicating an increase in the optical band gap [1,18].

Tauc's plots were constructed to determine the optical band gap values [1,11]. The band gap increased from approximately 1.45 eV for undoped CuO to higher values with increasing Mn concentration [1,9,19]. This blue shift in the band gap can be explained by the Burstein-Moss effect or by the introduction of structural defects that modify the electronic band structure [1,20].
The increase in band gap with Mn-doping is consistent with previous reports in the literature [1,21]. This optical property tuning is significant for optoelectronic applications where precise control of the band gap is required [1].

3.4 Electrical Properties

3.4.1 Dielectric Properties

The dielectric constant and dielectric loss of the films were studied as a function of frequency and Mn concentration [2]. The dielectric constant increased with increasing Mn concentration [2]. This enhancement can be attributed to the increased number of charge carriers and defects introduced by Mn-doping, which contribute to polarization mechanisms in the material [2,22].

The AC conductivity was calculated from the dielectric data using the relation $\sigma\_AC = 2\pi f\varepsilon_0\varepsilon'\tan\delta$ [2]. The AC conductivity curves showed an increasing conductivity value with increasing Mn content [2,5,23]. This enhancement in conductivity is due to the Mn doping, which induces defects at grain boundaries, facilitating charge transport [2].

The frequency dependence of conductivity followed Jonscher's universal power law, typical of disordered materials [2,24]. The observed behavior indicates that hopping of charge carriers between localized states is the dominant conduction mechanism [2,25].

4. Discussion

The results obtained in this study demonstrate the significant influence of Mn-doping on the structural, optical, and electrical properties of CuO nanoparticles synthesized by the SILAR method [1,2].

The structural analysis reveals that Mn ions successfully substitute Cu ions in the CuO lattice, as evidenced by the shift in XRD peak positions and changes in lattice parameters [2]. This substitution creates lattice strain and defects, which are reflected in the decrease in crystallite size with increasing Mn concentration [2,14]. The monoclinic structure is maintained for all doping levels, indicating that the crystal structure is stable upon Mn incorporation [1].

The optical properties show a clear trend of increasing band gap with Mn-doping [1,9]. This blue shift suggests that Mn-doping effectively modifies the electronic structure of CuO [1]. The incorporation of Mn ions introduces additional energy levels that hybridize with the CuO band structure, leading to a widening of the band gap [1,26].

The electrical properties exhibit enhancement with Mn-doping [2]. The increase in dielectric constant and AC conductivity can be attributed to the generation of charge carriers and defects at grain boundaries [2,5]. Mn ions in different valence states (Mn^{2+} and Mn^{3+}) create a mixed valence system that facilitates charge hopping and enhances electrical transport [2,27].

The improved properties observed in this study make Mn-doped CuO nanoparticles promising candidates for various applications [1,2]. The tunable band gap is advantageous for photodetector and solar cell applications where specific band gap values are required [1,28]. The enhanced conductivity and

dielectric properties are beneficial for microelectronic devices such as capacitors and memory devices [2,29].

The SILAR method demonstrated in this work offers advantages over other deposition techniques, including simplicity, low cost, and low temperature processing [1,30]. This makes it suitable for large-scale production of Mn-doped CuO films with controlled properties [1].

5. Conclusion

This research successfully synthesized undoped and Mn-doped CuO nanoparticles with plate-like nanostructures on glass substrates using the SILAR method [1]. The structural characterization confirms the monoclinic structure for all films, with successful incorporation of Mn ions into the CuO lattice [1,2]. The decrease in crystallite size from 123 nm to 45 nm with increasing Mn concentration indicates the influence of Mn-doping on crystal growth [2].

The optical studies reveal an increasing band gap with Mn-doping, demonstrating that the optical properties can be tuned by controlling the doping concentration [1]. The electrical characterization shows enhanced dielectric constant and AC conductivity for Mn-doped samples, attributed to defects and charge carriers introduced by Mn incorporation [2].

The results demonstrate that the SILAR method is an effective technique for producing Mn-doped CuO films with controllable properties [1]. The enhanced structural, optical, and electrical properties of Mn-doped CuO nanoparticles make them promising candidates for optoelectronic and microelectronic device applications [1,2].

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

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Ethical Approval:
This study does not involve any human participants or live animal experimentation. Therefore, ethical approval was not required for this research

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