

Effect of Mg Doping Level on the Physical Properties of CuO Nanoparticles and Fabrication of p-MDC/n-Si Junction Diodes for Optoelectronic Applications

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Abstract: Magnesium doping offers a route to modify the structural and optical properties of copper oxide (CuO) nanoparticles for optoelectronic devices. This study investigates the influence of Mg concentration on CuO nanoparticles and evaluates a p-type Mg-doped CuO/n-type silicon (p-MDC/n-Si) heterojunction photodiode. Undoped and Mg-doped CuO nanoparticles containing 1, 3, 6, and 9 wt% Mg were synthesized by coprecipitation. Their structural, morphological, elemental, vibrational, and optical properties were characterized using X-ray diffraction, field-emission scanning electron microscopy, energy-dispersive X-ray spectroscopy, Fourier-transform infrared spectroscopy, ultraviolet-visible diffuse reflectance spectroscopy, and photoluminescence measurements. X-ray diffraction confirmed a monoclinic CuO phase, with the smallest average crystallite size of 27 nm obtained at 3 wt% Mg. Electron microscopy revealed aggregated grains with varying morphologies, while elemental analysis detected Cu, O, and Mg. Optical measurements indicated Mg-dependent changes in absorption and estimated band gaps ranging from 1.3 to 2.5 eV. Infrared spectra exhibited Cu–O vibrations at 474 and 606 cm⁻¹, and photoluminescence peaks occurred at 364, 383, and 414 nm. Nanoparticles containing 9 wt% Mg were selected for diode fabrication. Current–voltage measurements demonstrated light-dependent electrical behavior, with the ideality factor decreasing from 6.8 in darkness to 3.9 under illumination. The diode exhibited 1085% photosensitivity and a reported specific detectivity of 2.1×10^9 Jones. These findings indicate the potential of Mg-doped CuO/silicon heterojunctions for photodetection, subject to further device validation and systematic measurements of stability and response speed.

Keywords: CuO Nanoparticle, Co-Precipitation, MDC NPs, I-V Characteristic, Detectivity, TEM analysis.

1. Introduction

In modern electronic world, the PN photodetector has widely used in numerous arenas, like deep UV light disinfection, optical communication, tracking, missile warning, fire detection, and fact switching applications due to its simple device structure, greatest sensitivity, long term life and rapid response [1-3]. Importantly, the external electric field should be applied to the device in order to enable charge separation of the photo-created electron-hole pairs (EHP). However, the built-in electric field in pn junction based photodetectors supports in the sufficient charge separation of photo-generated EHP, which results making them faster response in comparison with to other types of detector [4]. The metal oxide semiconductors like ZnO, CuO, and SnO

based PN diode have fascinated much attention due to their great breakdown electric field, optical absorbance, more saturation electron velocity, constant chemical properties, less resistivity and narrow band gap [5-7]. Consequently, pn heterojunction based photodetectors with metal oxide NPs have been extensively studied and utilized numerous applications. Among the metal oxide, the CuO NPs are the most promising candidate for advanced applications including optical sensor, switching, remote control, fire detection, battery and gas detection due to its outstanding properties like high surface to volume ratio, good electrical conduction, optical absorption, smaller particle size, low toxicity and narrow band gap [8-10]. CuO NPs have optical band gap approximately 1.2-2 eV depending upon the preparation technique and

precursor used. It can be formed with different oxidation states such as Cu^0 , CuI , Cu^{II} and Cu^{III} [11]. Compared to The CuO NPs are good inorganic material and more stable relative with organic NPs. Recently, Emad H. Hussein *et al.* [12] fabricated CdS/CuO/Cu nano-heterostructures by electrochemical route and obtained ideality factor more than unity. Jhansi *et al.* [13] developed p-CuO/n-Si and p-Al:CuO/n-Si diodes by spray pyrolysis method and achieved diode n value higher than unity. Rajeev R. Prabhu *et al.* [14] investigated p-CuO/n-ZnO heterojunction by sol-gel spin-coating technique and reported with a turn on voltage of 2.5 V.

Nevertheless, there is a challenging in the development of monoclinic p-type CuO NPs for the practical application of p-n photodetectors. The formation of junction with p-type semiconductors like monoclinic CuO NPs allowing to improve the built-in electric field for quick separation of photo-generated EHP [15]. Compare to n-type semiconductors, the p-type have direct transitions from heavy to light hole bands at the valence band. Thus, p- type carrier absorption is stronger in comparison with n-type. Moreover, the addition of metal ions can tuning the electrical conductivity resulting increase the forward current of the diode. The turn-on voltage of PN junction diode is comparatively larger than that of type diodes. Thus, the incorporation of metal doping into the host material can improve the forward conduction ability which are needed to balance the high turn-on voltage. The volunteer inclusion of metal dopant in to the host lattice contributing free electrons and permitting for the development of superiorly conductive p-type semiconductor layers. It was aided for the development of novel photodetector including PN junction and Schottky barrier diodes [16].

It is well known that the CuO can be prepared by several methods such as sputtering, spray pyrolysis, hydrothermal, co-precipitation, and spin-coping [17-20]. However, the co-precipitation method is promising candidate to prepare CuO NPs. Many parameters can be tailoring by this method including crystallite, surface morphology and energy gap. The precipitation method offers unique surface structures such as nanowire, nanorod, nanoneedle, nano-flower and nanoplate. The precipitation methods having several advantages like high purity, minimum time to make a preparation, low temperature, low-cost and not needed any organic fuels for this method. In recent, Bodrun Nahar *et al.* [21] Synthesized CuO NPs with spherical morphology by chemical precipitation and studied its Photocatalytic and antibacterial activities. Justin Paul *et al.* [22]

fabricated photodiode and studied its I-V characteristics by preparing CuO NPs by precipitation method.

The goal of the present work is to fabricate p-MDC/n-Si and analyse their I-V characteristics. To achieve the aim, we have prepared CuO NPs with different Mg doping level such as 0, 1, 3, 6 and 9 wt% by precipitation method and carefully studied their physical properties like structural, optical, morphological and electrical using XRD, FESEM with EDX, FTIR, UV-DRS, PL and I-V measurements.

2. Experimental Section

2.1 Materials

The chemicals used in this present work such as copper (II) nitrate trihydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$), magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) and Sodium hydroxide (NaOH) solution were purchased by sigma Aldrich with 99.99% purity.

2.2 Preparation of Bare CuO and Mg Doped MgO NPs

The CuO NPs have been fabricated by simple co-precipitation technique utilizing ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) and $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. Initially, 0.1 M of ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) was dissolved in 100 ml deionizing water and serried one hour by magnetic starrier. After that, the NaOH solution was added drop by drop until pH reached as 11. Favourably, a precipitates was obtained after pH level of 11. Then, the precipitates carefully washed three times by ethanol and deionizing water. The obtained precipitates were dried 80 °C using hot-air oven for 24 hours. Finally, the product was calcined at 400 °C for four hours by muffle furnace. To prepare Mg doped MgO NPs, the $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ was added as 1, 3, 6 and 9 wt% in the prepared 0.1 M of ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) solution separately. The same procedure was used to make Mg doped MgO NPs.

2.2 The p-MDC/n-Si Junction Diode Fabrication and Characterizations

The one side polished n-type silicon (Si) substrates used for the fabrication of p-MDC/n-Si junction diode. The Si substrate size approximately 1 cm x 1 cm and resistivity of 0.005 – 100 Ω -cm. Before diode fabrication, the Si substrate was cleaned properly with various steps using ethanol and Piranha solutions. To deposit p-type Mg@CuO layer, the previously prepaid un-doped and Mg-doped NiO NPs were dissolved in 5 mL of ethanol 9diluted), and added few

drops of HCL separately. The formed homogeneous solution was sensibly drop-casted on a well-cleaned n-type Si substrates using a facile drop-casting technique. The fabricated p-MDC/n-Si diode were annealed at 400 °C under for 1 hour. The silver adhesive was applied with copper wire on both surface of the prepared device for the electrical contact propose. After 3 hours the I-V studies of the diode was measure in room temperature.

The phase identification and crystallite size of the MDC NPs were studied by Powder X-Ray Diffractometer (X' Pert Pro – PANalytic) with wavelength of $\lambda = 1.54060 \text{ \AA}$. The FESEM and EDAX were carried out using SIGMA HV – Carl Zeiss with Bruker Quantax 200 – Z10 EDS Detector. Diffused reflectance Spectrophotometer (DRS) with model number UV-3600 from Shimadzu was used to analyse the optical characteristics of Mg-doped NiO NPs. The functional group and vibrational modes of the samples was studied by FTIR spectroscopy using Thermo Nicolet 380 FTIR spectrophotometer. The emission peaks of the obtained NPS was recorded by JASCO FP-8300 spectrofluorometer. The diode characteristics and photo-response of the devices were investigated by Keithly electrometer (6517/B) with portable solar simulator (PEC L01).

3. Result and Discussions

3.1 X-Ray Diffraction Analysis

The XRD pattern of pure CuO was compared with MDC NPs prepared by co-precipitation method with different doping levels such as 1.3, 6 and 9 wt% of Mg which are shown in Figure 1. The observed XRD peaks in pure CuO NPs and MDC NPs were indexed as

(110), (002), (111), (202), (020), (202), (113), (311) and (004) plane. Each XRD patterns are well matched with JCPDS file No. 45-0937 suggesting the monoclinic phase of the prepared MDC NPs [23]. No peaks and phase was detected related to Mg dopant. From Fig. 1, it is observed that each samples are polycrystalline in nature. There are two main peaks was recorded along the (002) and (111) direction conforming the formation of CuO NPs. At the same time, the intensity of these planes was decreased while adding Mg doping on CuO system particularly for 3 wt%. The reduction of intensity's in the Mg doped samples is might be due the change in electron density or point defects and lattice distortion [24]. The ionic radius of magnesium $\text{Mg}^{2+}(0.65 \text{ \AA})$ has comparably lesser than copper $\text{Cu}^{2+}(0.99 \text{ \AA})$ [23]. No peak shifting was detected in the Mg doped samples. It is obvious that the doping element successfully substituted in to the CuO lattice without changing their crystal structure. Debye-Scherer's equation (1) was used to estimate the crystallite size of the prepared NPs.

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

Where, k is shape factor (0.9), λ is wavelength of X-ray ($1.5406 \text{ \AA}$), β is with FWHM and θ is diffraction angle. The estimated average crystallite size (D_{avg}) of un-doped NPs is 41 nm. Interestingly, as the Mg doping level increases, the D_{avg} values of the doped samples are decreased gradually up to 3 wt% and somewhat increased for 6 and 9 wt% which is listed in Table 1. The 3 wt% showed minimum D_{avg} of 27 nm compared to other doping levels. This can be attributed to the dissimilarity of ionic radius of Mg and Cu element's [23, 24].

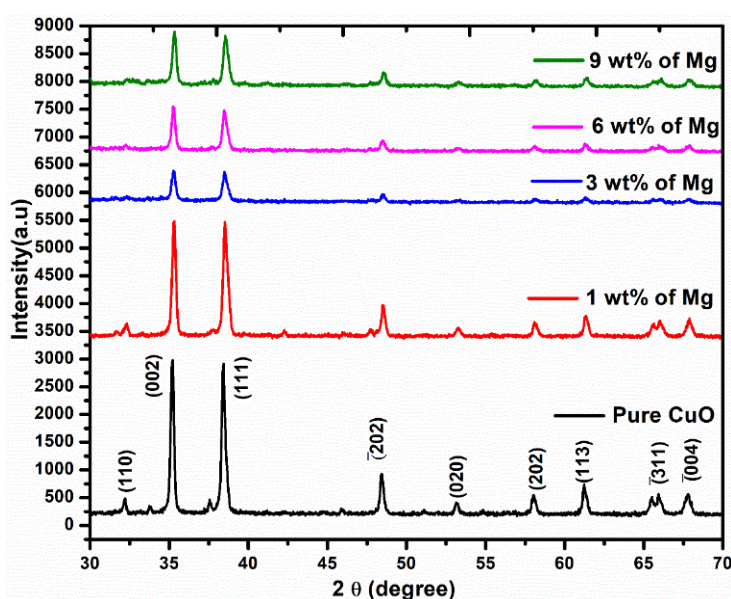


Figure 1. XRD profile of bare CuO and MDC NPs for various concentrations.

**Table 1.** structural parameters of pure CuO and MDC NPs for different Mg concentration

Samples	Average Crystallite Size (D_{avg})	Dislocation density	Micro-strain	Stacking Fault
Pure CuO	41	5.72×10^{14}	0.000829	0.0022
1 wt% of CuO	29	1.29×10^{15}	0.001229	0.0034
3 wt% of Mg	27	1.38×10^{15}	0.001277	0.0035
6 wt% of Mg	28	1.31×10^{15}	0.001244	0.0034
9 wt% of Mg	29	1.21×10^{15}	0.001194	0.0033

The calculated other micro-structural parameters like micro-strain (ϵ), dislocation density(δ) and stacking fault (SF) found to be reduced continuously while increasing Mg concentration from 0 to 3 w% (see Table 1) and increased slightly for 6 and 9 wt% which is due to the improvement of crystallite size of CuO NPs after Mg doping. The obtained minimum crystallite size exhibited higher microstrain of 0.001277. The microstrain mainly attributed to the non-uniform lattice distortions, antiphase domain boundaries, dislocations, grain surface and faulting [25]. The dislocation density changed from 5.72×10^{14} to 1.38×10^{15} with Mg doping levels. The variation of ionic radius of doping and host elements greatly influence the dislocation density of the samples because the doping of Mg ions with different atomic radii may generate lattice strain in the nanocrystals and that results in structural distortion [26]. Moreover, the decline of strain and dislocation density can be accredited to the increment of crystallinity of the NPs due to reduction of lattice misfit in the NPs.

$$\epsilon = \frac{\beta}{4 \tan \theta} \quad (2)$$

$$\delta = \frac{1}{D^2} \quad (3)$$

$$SF = \left[\frac{2\pi^2}{45(3 \tan \theta)^2} \right] \beta \quad (4)$$

Where, θ is a peak position and β was full width at half -maximum (FWHM) δ is a micro-strain and D is crystallite size.

3.2 FESEM and EDX Analysis

Both surface morphology and elemental composition of the synthesized NPs were examined by FESEM and EDX analysis. Figure 2a-c displays the FESEM images of 9 wt% of MDC NPs for various magnification like (a) 1 μ m (b) 200 nm (c) 100 nm. From FESEM images, It was noticed that the most of the grains, which are nanoscale in range.

The obtained nanoparticles were aggregated on the surface. These results could have originated from the high surface energy of the prepared MDC NPs. It also recorded that each crystal grains are arranged different in shape and size. The difference in the shape and size of the NPs is mainly due to the doping element (Mg) on the CuO system. Also, the weak inter-intra particle forces is possible reason for changes of grain size and their shape of NPs. The EDX spectra of 9 wt% of MDC NPs is shown in Figure 2d. The expected elements including oxygen (O), Mg and Cu were observed. The atomic % of Cu=40.82 %, O=56.45% and Mg=2.73%. No other elements was detected. These result suggested that high-purity of the prepared samples.

3.3 UV-DRS Spectrum

The absorbance spectrum of MDC NPs was compared with pure CuO which is shown in Figure 3. From the absorbance spectrum, a broad absorption band was recorded among the wavelength range from 250 and 850 nm. This could be attributed to the black colour of the prepared MDC NPs that absorb in the whole region. With the addition of Mg ions as a dopant onto a CuO system, a significant variations in absorption intensity was detected, demonstrating the influence of Mg incorporation on the optical properties of CuO. The absorption intensity all doped samples lesser than that of non-doped CuO. However, the 3, 6 and 9 wt% showed increment in absorption intensity relatively with 1 wt% due to increase in oxygen vacancies. The energy of optical band gap values for un-doped CuO and Mg doping CuO NPs have been determination by Tauc's plot using the following equation.

$$(\alpha h \nu)^2 = A(h\nu - E_g) \quad (5)$$

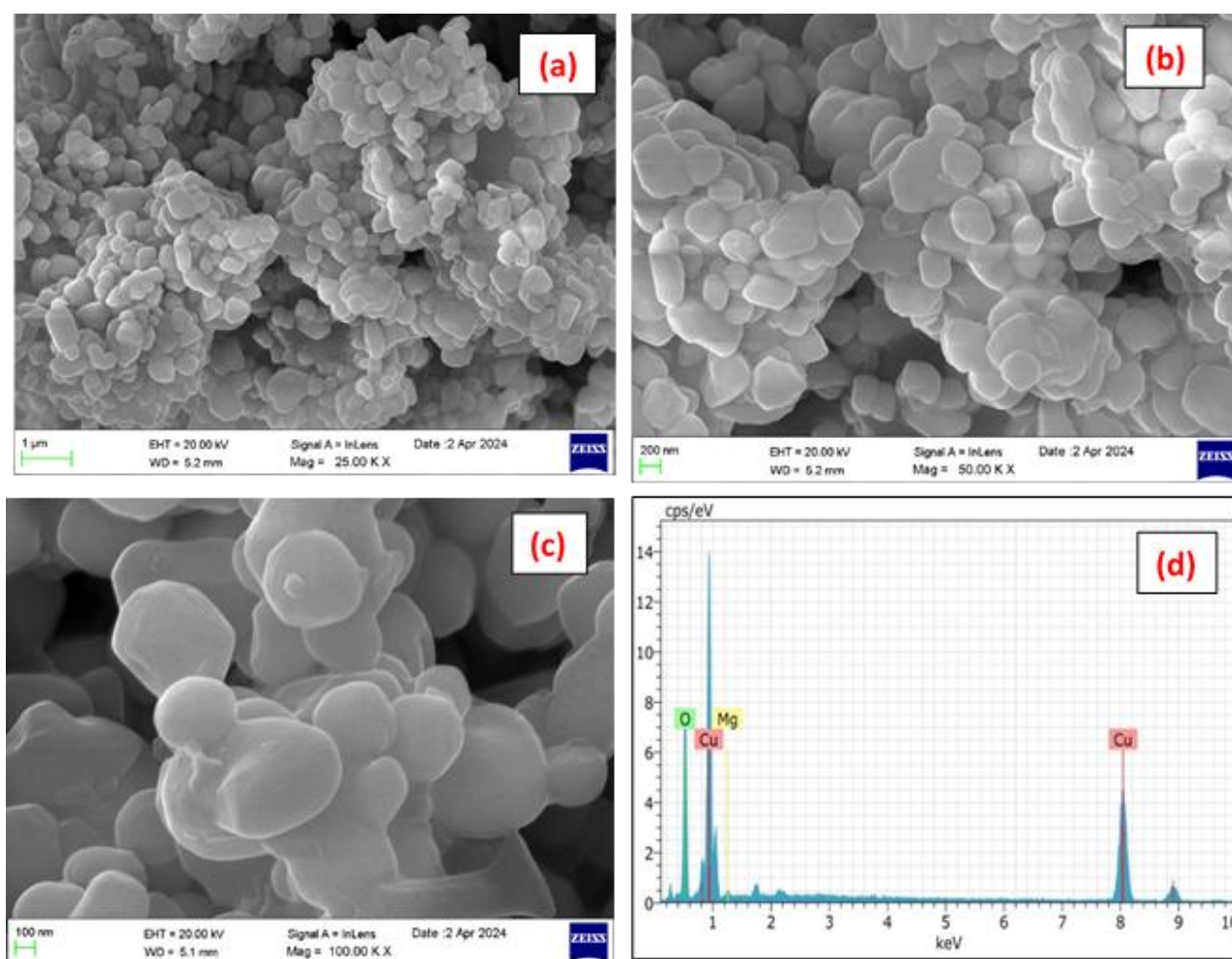


Figure 2. FESEM images of 9 wt% of MDC NPs for (a) 1 μm (b) 200 nm (c) 100 nm (d) EDX spectra for 9 wt% of MDC NPs.

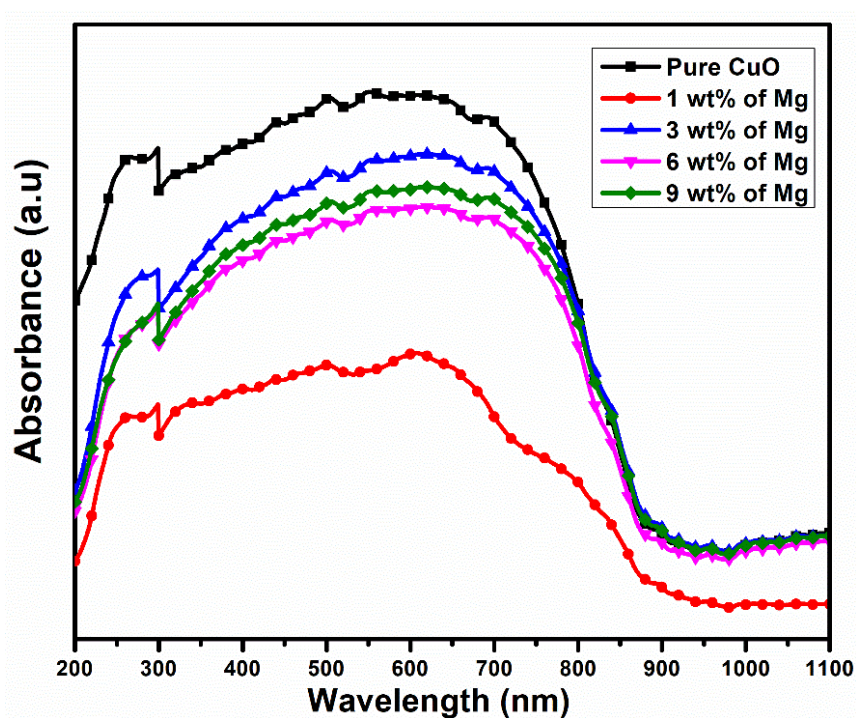


Figure 3. Absorbance spectrum of bare CuO and MDC NPs with different doping levels.

The calculated E_g values for bare CuO, and 1, 3, 6, and 9 wt% MDC NPs are found to be 1.3, 2.0, 2.5, and 2.2 eV, respectively. These values are good accordance with earlier reports [23, 24]. Compared to pure CuO, the estimated band gap value of Mg doped samples increases with increasing Mg doping concentration from 0 to 9 wt% as depicted in Figure 4, which is accredited to the Burstein-Moss effect. At the same time, the band gap of 1, 6 and 9 wt% reduced compared to 3 wt% of Mg, which can be explained by the band gap renormalization effect. The reduced band

gap enables to improve the photon absorption process and also electron excitation [27]. The inclusion of Mg ions favourably alters the absorption intensity and bandgap of the synthesised CuO NPs.

3.4 FTIR Analysis

The FTIR spectrum was utilized to determine the functional group of un-doped and MDC NPs which are shown in Figure 5.

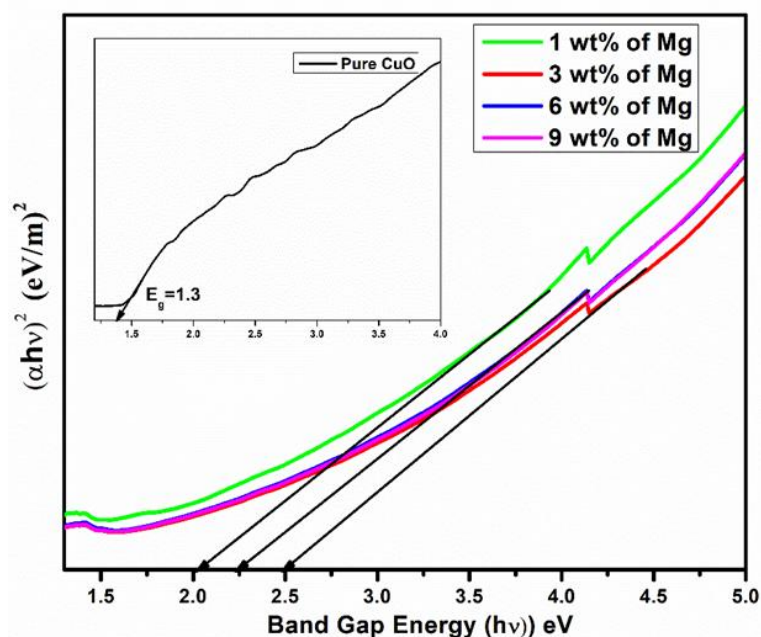


Figure 4. Band gap values of bare CuO and Mg doped MgO NPs with different doping levels.

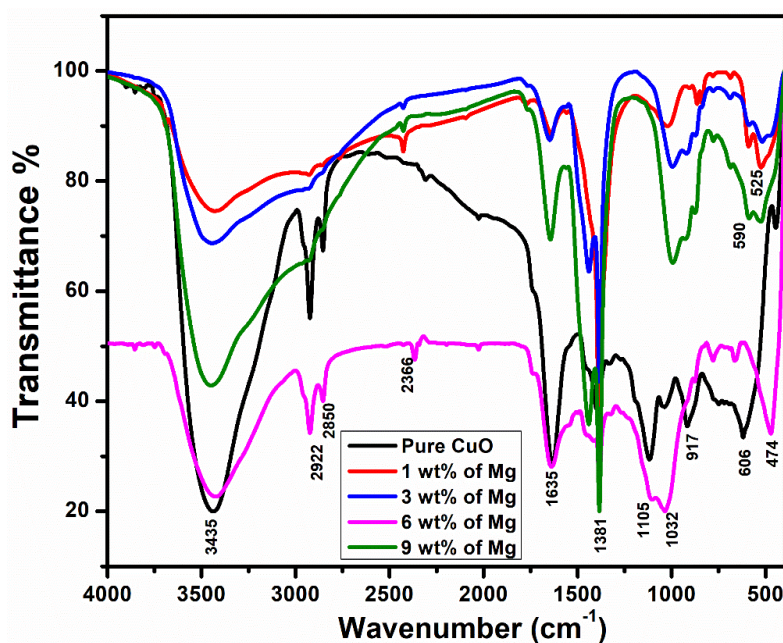


Figure 5. FTIR spectra bare CuO and Mg doped MgO NP with different doping levels.



Each functional groups which were formed in un-doped CuO NPs are also obtained in MDC NPs. The FTIR peaks observed at 474, 606, 917, 1032, 1105, 1381, 1635, 2366, 2925, 2922, 3435 cm^{-1} respectively. These are aligns well with reported values [28, 29]. It is obvious from the FTIR spectra, the Cu–O bond was observed at 474 and 606 cm^{-1} . In general, the peaks before a wavenumber of 1000 cm^{-1} are related with metal interactions. The CuO NPs revealed the occurrence of deformation vibration of the C–H group of alkanes at 2922 cm^{-1} . Moreover, the peaks around 3435 and 1635 cm^{-1} can be associated to the O–H stretching vibrations adsorbed on the surface of the CuO [28, 29]. These result shows the successful calcination of the CuO NPs. The additional peaks observed 590 and 525 cm^{-1} in the 1, 3, and 6 wt% of Mg doped samples was due to Mg–O, indicting successful doping of Mg metal on the surface of pure CuO NPs.

3.5 Photoluminescence Spectrum

The PL spectrum of the un-doped and MDC NPs were recorded among the wavelength of 330 and 450 nm which is depicted in Figure 6. Specifically, double emission peaks were recorded in the UV region at 364 nm and 383 nm for all the samples. At the visible region, a violet emission peak was obtained

around 414 nm due to the electronic transition of the interstitial atoms, oxygen vacancies, and also grain boundaries in the NPs [30]. Addition of Mg ions into the CuO NPs could not be affected the peaks of position. But, affecting their intensity because the Mg dopant improves the emission peak intensity's of the CuO NPs. The Mg doping level increases crystallinity and decreases non-radiative recombination, resulting improves PL intensity of CuO NPs [23].

3.6 The p-MDC/n-Si Junction Diode Characteristics

Based on the previous characteristics of the prepared NPs, it is observed that the 9 wt% of MDC NPs exposed unique results including crystallite size, surface structure and band gap. Thus, the 9 wt% of MDC NPs was used to fabricate p-n junction diode and analysed their I-V characteristics for dark and light environment which is shown in Figure 7. Compared to dark, diode measured under light condition exhibited better current values particularly at forward bias due to the creation of large photo-carrier on the MDC NPs surface suggesting the photo-conducting nature of the present diode [31, 32]. The current transportation of the p-MDC/n-Si junction diode was studied by thermionic emission model using the following equations [31, 32].

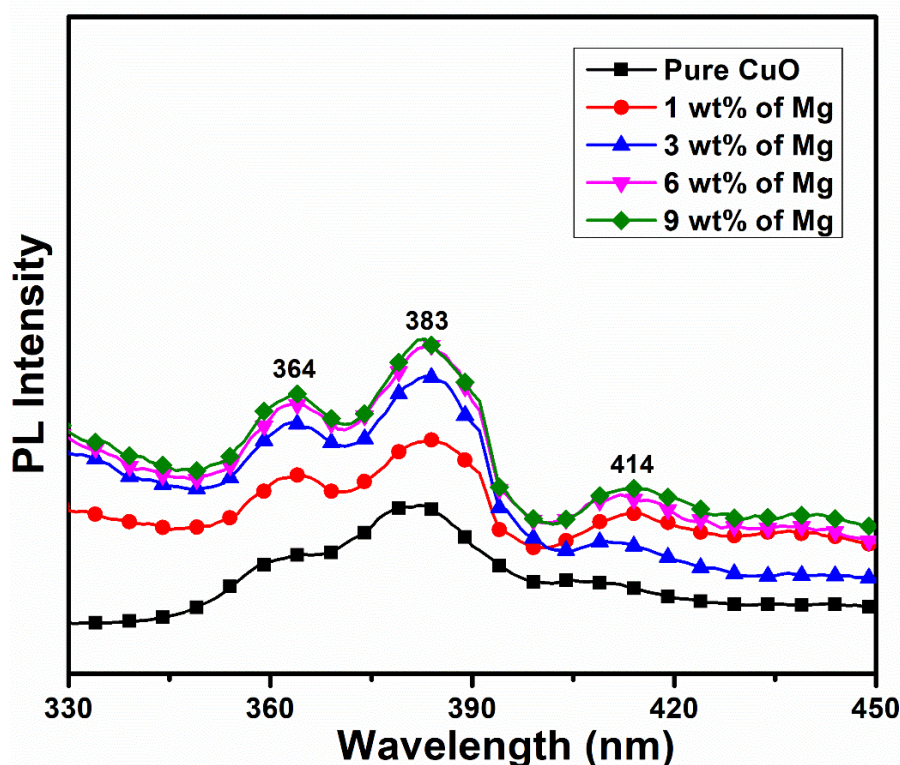


Figure 6. PL spectrum of pure CuO and MDC NPs for various doping level.

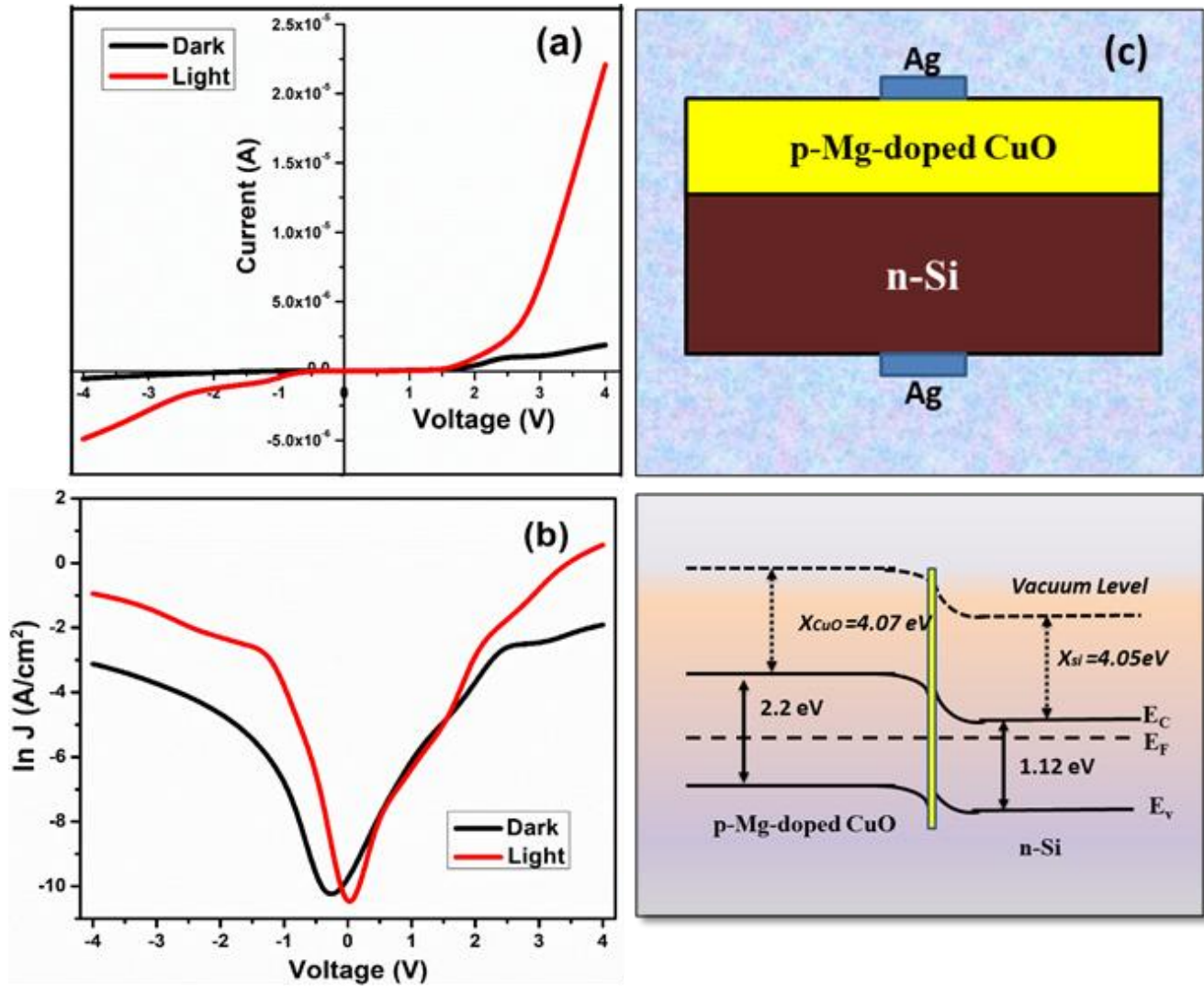


Figure 7. (a) I-V characteristics (b) J-V plot (c) Schematic diagram (d) energy level diagram of p-MDC/n-Si for dark and light environment.

$$J = J_0 \exp\left(\frac{qV}{nK_B T} - 1\right) \quad (6)$$

$$n = \frac{q}{k_B T} \left(\frac{d(V)}{d(\ln(J))} \right) \quad (7)$$

$$\Phi_B = \frac{k_B T}{q} \ln\left(\frac{A A^* T^2}{J_0}\right) \quad (8)$$

Where J is the current density (A/cm^2), A is the area of the diode ($1 \text{ cm} \times 1 \text{ cm}$), A^* is the Richards constant ($112 \text{ cm}^{-2} \text{ K}^{-2}$) Φ_B is the barrier height, J_0 is the leakage current density (A/cm^2), q is the electron charge, V is the bias voltage, n is the ideality factor, Φ_B is the effective barrier height, K_B is the Boltzmann constant and T is the temperature.

The diode ideality factor calculated as 6.8 for dark condition, which was favourably decreased as 3.9 under light environment. This result signifying that the ideality factor of the MDC NPs is sensitive for light. The reduction of n values is due to the self-formation of charge couples on the p-type semiconductor layer. Also, less recombination, tunnelling current, and

separation of charge carrier [33, 34]. The estimated barrier height values are 0.81 and 0.79 eV for dark and light environment. The variation in the obtained barrier height might be the applied voltage and diffusion of minority carriers in the reverse bias condition. The determined values of n and Φ_B of the present work is well agreed with previous literature [34-35]. The photodetector properties of the MDC NPs was studied by the following equations [36].

$$(P_s) = \left(\frac{I_{ph} - I_D}{I_D} \right) \times 100\% \quad (9)$$

$$(R) = \left(\frac{I_{ph}}{P A} \right) \quad (10)$$

$$(QE) = \left(\frac{R h c}{q \lambda} \right) \quad (11)$$

$$(D^*) = \left(\frac{R}{2 q I_D} \right) \quad (12)$$

Where I_{ph} is the photocurrent, I_D is the dark current. P is the irradiation of the lamp and A is the area of the diode h is the Planck's constant, R is the

responsivity, c is the light velocity, q is the charge of e^- and λ is the wavelength. The p-MDC/n-Si diode disclosed photosensitivity and responsivity of 1085 % and 6.3 W/cm² respectively. Figure 7 (c and c) represents the Schematic diagram and energy level diagram of p-MDC/n-Si junction for dark and light environment. Photosensitivity and responsivity of the diode is associated with enhancement of quantum efficiency, absorption coefficient, electric field strength at depletion area and carrier mobility in the prepared NPs [37]. As expected, the p-MDC/n-Si diode exposed quantum efficiency and detectivity of 2 % and 2.1×10^9 Jones. The observed QE value confirming the formation of higher charge carriers under light conditions. Similarly, the D^* provides the information of the ability of light that can be detected even weak optical signal. The higher D^* values implies that the better device performance of the present diode [38, 39]. The calculated QE and D^* of the p-MDC/n-Si diode are good accordance with earlier literature [38-40]. The calculated photodiode parameters of p-MDC/n-Si diode suggested various optoelectronic applications including photo-sensor, solar cell, photo-detector and photo-transistor.

4. Conclusion

We have successfully fabricated p-MDC/n-Si diode using chemically synthesised MDC NPs. The structural, morphological, optical and electrical properties of the MDC NPs were studied by various futures techniques like XRD, FESEM with EDX, FTIR, UV-DRS, PL and I-V characteristics. A single phase monoclinic phase was observed from the XRD pattern with the minimum D_{avg} of 27 nm for 3 wt% Mg doped NPs. From FESEM picture, a randomly arrange crystal grains which are aggregated on the surface. The doping and host elements with oxygen were detected in the EDX spectrum. The functional group of the prepared was analysed by FTIR spectrum. In UV-DRS, the higher doping of 9 wt% sample showed maximum E_g values of 2.5 eV. A three emission peaks related to violet and blue was recorded from the PL spectrum. The diode cure conforming the well rectification nature of the present diode. The minimum n values of 3.9 obtained for under light environment which is less than that of dark. The sensitivity and quantum efficiency of the fabricated device are 1085 % and 2 % respectively. Overall, we have concluded that the p-MDC/n-Si diode is most appropriate for light detection application.

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Author Contribution Statemen

P. Rajapandi: Conceptualization, Methodology, Writing - Original Draft. G. Viruthagiri: Formal Analysis, Writing - Original Draft. N. Sumathi: Data Curation, Writing - Review & Editing. M. Vidhya: Writing - Review & Editing. A. Ajith: Writing - Review & Editing. Bharathi Raja: Writing – Review & Editing. All the authors read and approved the final version of this manuscript.

Does this article screened for similarity?

Yes

Conflict of interest

The Author's declares that there is no conflict of interest anywhere.

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