Article

Synthesis and Optical Properties of B-Mg co-Doped ZnO Nanoparticles

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Abstract: Doping impurity into ZnO is an effective and powerful technique to tailor structures and enhance its optical properties. In this work, $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ and $\text{Zn}_{1-x-y}\text{Mg}_x\text{B}_y\text{O}$ nanoparticles ($x = 0, 0.1, 0.2, 0.3, 0.4$; $y = 0, 0.02, 0.04$) were synthesized via one-pot method. It shows that the Mg and B dopants has great influence on crystallinity and surface morphology of ZnO nanoparticles, without changing the wurtzite structure of ZnO. The band structure study indicates that the competition of Conductive Band (CB) shift, Burstein–Moss (B-M) shift and Shrinkage effect will cause the band gap energy change in ZnO.

Keywords: one-pot method; B-Mg co-doped ZnO; crystal structure; optical band gap

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1. Introduction

As one of the third-generation wide band gap semiconductor materials, zinc oxide (ZnO) has attracted great attention in the field of optoelectronic functional materials due to its excellent physical and chemical properties, such as large exciton binding energy (60 meV), wide direct band gap energy (3.37 eV), low cost, and facile preparation [1–4]. In recent years, researchers have discovered that ZnO nanoparticles (NPs) have unique and superior structural, optical, electronic, and chemical properties compared to bulk ZnO materials [5,6]. Therefore, various methods for preparing ZnO NPs have been developed, such as co-precipitation, hydrothermal, combustion, spray pyrolysis, sol-gel, and high-temperature pyrolysis [7–11].

Doping impurity elements into the target lattice is an effective and powerful technique that can change the electronic structure and enhance electrical and optical properties [12]. Various elements (for example, Al, Ga, Mg, Mn) [13–16] have been used as donors to improve the optical, physical, and chemical properties of ZnO. Among them, Mg-doped ZnO NPs have attracted great attention; because the ionic radius between Mg^{2+} and Zn^{2+} is equivalent, doping Mg element will not cause huge lattice changes. Moreover, because of the large band gap of MgO, Mg doping can broaden the optical band gap of ZnO [17,18]. Jongchul Seo [19] synthesized Mg-doped ZnO ($\text{Zn}_{1-x}\text{Mg}_x\text{O}$, where $x = 0.000–0.010$ M) nanoparticles via a co-precipitation method and showed an increase in the optical band gap from 3.32 to 3.51 eV with respect to the increase in the concentration of Mg doping. Riffat Sagheer [5] synthesized $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ nanoparticles ($x = 0–0.1$, $\Delta x = 0.02$) via a co-precipitation method; the E_g of Mg-doped ZnO nanoparticles (3.080 eV) is greater than that of undoped ZnO nanoparticles (3.038 eV). XRD results confirmed that the synthesized nanoparticles are polycrystalline, which have a typical hexagonal wurtzite structure, and there are no other impurities or dopant phases in the ZnO crystal lattice. Charis Caroline [20] synthesized pure ZnO and Mg doped ZnO ($\text{Zn}_{1-x}\text{Mg}_x\text{O}$; $x = 0, 0.2, 0.4$) by sol-gel method. The results show that the E_g increases from 3.16 to 3.24 eV with the Mg concentration from 0 to 0.4.

In addition, the group III elements are usually doped to increase the electrical properties of ZnO by increasing the carrier concentration [21]. Al is considered to be the best doping element to enhance optical and electrical properties [22]. B element is also used to investigate the transparent and electrically conductive of ZnO and found that the transmittance of B-doped ZnO thin films were obviously improved [23,24], but the optical band gap was decreased [21].

Although lots of research was carried out on the elements doping in ZnO, the effect of B-Mg co-doped was rarely seen. Therefore, in this work, pure ZnO, Mg-doped ZnO, and B-Mg co-doped ZnO NPs were synthesized by simple one-pot method. The effect of Mg and B doping on crystal structure, morphology, and the band structure of ZnO will be investigated.

2. Materials and Methods

2.1. Synthesis of Nanoparticles

For the present research work, $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ and $\text{Zn}_{1-x-y}\text{Mg}_x\text{B}_y\text{O}$ nanoparticles ($x = 0, 0.1, 0.2, 0.3, 0.4$; $y = 0, 0.02, 0.04$) were synthesized via one-pot method. Here Zinc acetylacetonate ($\text{Zn}(\text{acac})_2$), Magnesium acetylacetonate ($\text{Mg}(\text{acac})_2$), Boric acid (H_3BO_3) were used as the Zn source, Mg source and B source, respectively. All the chemical ingredients were weighted in stoichiometric proportions for getting 10 mmol of final product. Then, a mixture of $\text{Zn}(\text{acac})_2$, $\text{Mg}(\text{acac})_2$, H_3BO_3 , oleic acid (OA, 0.9 g), oleylamine (OAm, 2.50 g), and octadecene (ODE, 13.2 g) with specific composition was degassed at 120°C for 30 min and heated to 280°C in a closed beaker. The hot mixture was cooled to room temperature naturally in the air, and then, 30 mL of ethanol was added to precipitate the nanoparticles, the mixture solution was centrifuged at 5000 r/min for 5 min to get white precipitates. The precipitates were redispersed in 10 mL of n-hexane and washed with ethanol for three times. Finally, the precipitates were dried in hot air oven at 100°C for 10 h. The dried precipitates were crushed to obtain desired nanoparticles.

2.2. Characterization of Nanoparticles

The crystal structure of all samples were characterized by X-ray diffraction with CuK α radiation (XRD, Rigaku, Tokyo, Japan). Analysis of surface features of all nanoparticles were carried out by using Sigma 500 Scanning electron microscope (SEM, Carl Zeiss AG, Jena, Germany) and Talos-F200s Transmission electron microscope (TEM, FEI, Waltham, MA, USA). Optical properties of the nanoparticles were explored by employing by a Scan UV spectrophotometer (UV-2700, Shimadzu, Tokyo, Japan). The UV diffuse reflectance spectroscopy were recorded at room temperature ranging from 200 to 800 nm.

3. Results

3.1. XRD Analysis

To investigate the structural aspects of the ZnO, Mg doped ZnO and B-Mg doped ZnO nanoparticles, XRD patterns of all samples were listed and analyzed. The XRD patterns of $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ nanoparticles with different doping concentrations ($x = 0, 0.1, 0.2, 0.3, 0.4$) synthesized by the one-pot method are shown in Figure 1a,b. It can be seen from the patterns that the synthesized nanoparticles have sharp diffraction peaks, which indicates that the prepared nanoparticles have good crystallinity. The characteristic diffraction peaks indicate that the synthesized nanocrystals are ZnO with a wurtzite hexagonal structure (PDF# 80-0074). Moreover, no characteristic peak corresponding to the “Mg” element or its compound was detected, which indicates that “Mg” is a dopant, and no additional Mg related extrinsic phase exists in the ZnO matrix. For the wurtzite phase, calculate the lattice constants “a” and “c” of (100) and (002), respectively, as follows [25].

Figure 1. XRD pattern of $\text{Zn}_{1-x-y}\text{Mg}_x\text{B}_y\text{O}$. (a) $\text{Zn}_{1-x}\text{Mg}_x\text{O}$; (b) enlarged XRD pattern of (100) and (002) plane of $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ in (a); (c) $\text{Zn}_{1-x-0.02}\text{Mg}_x\text{B}_{0.02}\text{O}$; (d) $\text{Zn}_{1-x-0.04}\text{Mg}_x\text{B}_{0.04}\text{O}$.

$$a = \frac{\lambda}{\sqrt{3} \sin \theta_{(100)}}, \quad (1)$$

$$c = \frac{\lambda}{\sin \theta_{(002)}}, \quad (2)$$

where λ is the wavelength of the incident X-ray ($\lambda = 1.5406 \text{ \AA}$), and θ is the Bragg angle. The influence of “Mg” atoms was investigated through the changes of peak positions of (100) and (002) planes, as in Figure 1b. Both peaks move to the lower 2θ value, as shown in the two circles in Figure 1b, indicating that the “Mg” atoms occupy the substitution lattice sites and interstitial sites [5]. However, the aspect ratio “ c/a ” listed in Table 1 is almost constant, which indicates that the dopant atoms are successfully combined with the ZnO host lattice without changing the crystal structure of the nanoparticles. In addition, according to Vegard’s law, a slight increase in the lattice parameters (“ a ” and “ c ”) of the ZnO lattice is noticed due to the high Mg doping concentration.

Table 1. Calculated parameters for nanoparticles.

Compounds	Dopant x	Lattice Parameters		a/c	Volume of Unit Cell V	Crystallite Size D (nm)	Dislocation Density	Strain
ZnO		3.244	5.198	1.603	47.366	38.885	6.614	8.914
$\text{Zn}_{1-x}\text{Mg}_x\text{O}$	0.1	3.247	5.204	1.603	47.510	46.441	4.637	7.464
	0.2	3.246	5.201	1.602	47.473	43.315	5.330	8.003
	0.3	3.252	5.212	1.603	47.750	42.863	5.443	8.087
	0.4	3.251	5.209	1.603	47.662	39.803	6.312	8.709
$\text{Zn}_{1-x-0.02}\text{Mg}_x\text{B}_{0.02}\text{O}$	0.1	3.245	5.200	1.601	47.497	43.090	5.386	8.044
	0.2	3.250	5.203	1.601	47.578	40.190	6.191	8.625
	0.3	3.248	5.201	1.602	47.510	39.247	6.492	8.832
	0.4	3.251	5.207	1.602	47.650	39.061	6.554	8.874
$\text{Zn}_{1-x-0.04}\text{Mg}_x\text{B}_{0.04}\text{O}$	0.1	3.250	5.204	1.601	47.596	39.999	6.250	8.666
	0.2	3.248	5.204	1.602	47.542	41.795	5.725	8.294
	0.3	3.250	5.204	1.601	47.596	39.248	6.492	8.832
	0.4	3.249	5.206	1.602	47.583	40.579	6.073	8.542

Besides, the average crystallite size D was calculated from XRD peak of (101) by using the Debye–Scherrer equation, the dislocation density δ is defined as the length of dislocation lines per unit volume of the crystal, which is determined from the crystallite size D . We can also calculate the strain ε caused by the crystal defects and distortion [25].

$$D = \frac{K\lambda}{\beta_{hkl} \cos \theta}, \quad (3)$$

$$\delta = \frac{1}{D^2}, \quad (4)$$

$$\varepsilon = \frac{\beta_{hkl} \cos \theta}{4}, \quad (5)$$

where β_{hkl} is the integral half width, K is a constant equal to 0.9. The calculated data are shown in Table 1. The crystal size of pure ZnO synthesized by one-pot method is only 38.9 nm, and it can be observed that the crystallite size increases while “Mg” atoms was doped, as shown in Table 1. Internal strain is generated in the parent ZnO system due to the difference between the ionic radii of Zn (0.057 nm) and Mg (0.060 nm). When the amount of doped Mg is small ($x = 0.1$), the doping atoms accelerate the diffusion rate in the Mg-doped ZnO nanoparticles due to the small volume of Mg^{2+} ions, resulting in a larger crystal size. The reason may be due to the low Pauling electro-negativity (1.31) and the high reactivity of Mg [5]. However, as presented in Table 1, the crystal size D of the sample decreases as the doping concentration increases ($x = 0.2, 0.3, 0.4$) due to the influence of lattice strain and dislocation density [26].

Figure 1c,d shows the XRD images of B-Mg co-doped ZnO nanoparticles with different B doping concentrations ($y = 0.02, 0.04$). Similar to Figure 1a, the addition of B element would not cause other additional peaks, indicating that B-Mg co-doping did not change the wurtzite structure of ZnO. However, the peak intensity decreases as the concentration of B increases, which indicates that the crystalline quality of the product is poor.

The differences between the crystallite size D of $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ and the one of B codoped $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ are presented in Figure 2a, to investigate the influence of B element. We have a medium D of 43.3 nm for $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ sample, while in presence of B medium, D is 40.3 and 40.5 nm. It seems that the Mg concentration is less effective on the variation of D when samples are co-doped with B. The reason may be that the incorporation of B element increases the lattice strain and dislocation density of zinc oxide nanoparticles [26], as shown in Table 1 and Figure 2b,c.

Figure 2. Comparison of calculated parameters of Zn_{1-x-y}Mg_xB_yO with different fraction of Mg. (a) crystallite size D; (b) dislocation density; (c) strain.

3.2. SEM and TEM Analysis

Using scanning electron microscope to study the effect of B and Mg doping on the surface morphology of ZnO nanoparticles, as presented in Figure 3. It can be observed that the pure ZnO synthesized by one-pot method has triangular and spherical grains and are seriously aggregated. When Mg doped in ZnO, as presented in Figure 3b–d, discal and hexagonal shapes appear, which means that the orientation of the sample is mostly perpendicular to the growth direction along the c-axis [27]. Besides, twinned crystal structures with a central vacant space in each twined building block have self-assembled. This phenomenon indicates that Mg element promotes the crystallization of ZnO. Comparing Figure 3d–f, it can be seen that with the addition of B element, the twin hexagonal crystal structure gradually disappears, and only a spherical structure exists in the Zn_{0.68}Mg_{0.3}B_{0.02}O sample in Figure 3f. The B element reduces the crystallinity of ZnO, which is in agreement with the result of XRD.

Figure 3. SEM images of pure and doped ZnO nanoparticles. (a) Pure ZnO; (b) Zn_{0.9}Mg_{0.1}O; (c) Zn_{0.6}Mg_{0.4}O; (d) Zn_{0.7}Mg_{0.3}O; (e) Zn_{0.68}Mg_{0.3}B_{0.02}O; (f) Zn_{0.66}Mg_{0.3}B_{0.04}O.

The typical transmission electronic microscopy (TEM) images of pure ZnO, $\text{Zn}_{0.7}\text{Mg}_{0.3}\text{O}$, $\text{Zn}_{0.68}\text{Mg}_{0.3}\text{B}_{0.02}\text{O}$, and $\text{Zn}_{0.66}\text{Mg}_{0.3}\text{B}_{0.04}\text{O}$ nanoparticles are presented in Figure 4. Pure ZnO nanoparticles with an average particle size of 20–50 nm are agglomerated. The well-resolved interfringe distance of 2.89 Å corresponds to the (100) lattice planes of ZnO. In the $\text{Zn}_{0.7}\text{Mg}_{0.3}\text{O}$ sample, discal-shaped particles showing good crystallinity are observed, which is consistent with the SEM results. After doping B ions, only spherical structures are present in Figure 4c,d. In addition, the lattice resolved images in Figure 4b–d all show that the lattice spacing is 2.85 Å, which can correspond to the (100) plane of ZnO, confirming that B-Mg co-doping did not change the structure of wurtzite zinc oxide.

Figure 4. Typical transmission electronic microscopy (TEM) images of (a) pure ZnO, (b) $\text{Zn}_{0.7}\text{Mg}_{0.3}\text{O}$, (c) $\text{Zn}_{0.68}\text{Mg}_{0.3}\text{B}_{0.02}\text{O}$, and (d) $\text{Zn}_{0.66}\text{Mg}_{0.3}\text{B}_{0.04}\text{O}$ nanoparticles.

3.3. UV–VIS Analysis

Ultraviolet-visible spectroscopy was used to study and analyze the optical properties of pure and B-Mg co-doped ZnO nanoparticles. Figure 5 presents the UV diffuse reflectance spectra of pure and Mg-doped ZnO nanoparticles with different Mg concentrations. It can be observed that there is a blue shift of the reflection peak between 370 and 400 nm. This is a phenomenon in which the band gap of a semiconductor increases, that is, the absorption is pushed to higher energy due to an increase in the population in the conduction band. On further observation, as shown in the enlarged image in Figure 5a, compared with pure ZnO, Mg-doped ZnO nanoparticles have higher reflectance, which is due to the increase in E_g .

Figure 5. (a) UV reflectance spectra of $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ nanoparticles; the inset is the enlarged figure in the red circle; (b) Tauc plots for determination of optical band gap energy of $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ nanoparticles.

The optical bandgap energy was calculated by the Kubelka–Munk equation [28], as follows.

$$F(R) = \frac{(1 - R)^2}{2R}, \quad (6)$$

where R is the reflectance. $F(R)$ is the absorption coefficient (α) in Tauc equation [28]:

$$(\alpha h\nu)^2 = A(h\nu - E_g), \quad (7)$$

We estimate the E_g by extrapolating the linear plot of the Tauc equation, as presented in Figure 5b. It can be observed that the E_g values increased from 3.286 eV to 3.318 eV for undoped ZnO and $\text{Zn}_{0.6}\text{Mg}_{0.4}\text{O}$ nanoparticles. It is because of the conduction band shift (CB shift) owing to the wider E_g of MgO (7.7 eV), as presented in Figure 6b. In addition, the E_g of $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ nanoparticles increase gradually with the increase of the Mg dopants. The reason may be due to the Burstein–Moss shift (B–M shift) caused by more electrons generated by substituting Mg^{2+} ions in ZnO. The schematic showing effect of CB shift and B–M shift is presented in Figure 6c.

Figure 6. Schematic showing effect of doping on ZnO band structure. (a) Band structure of pure ZnO; (b) CB shift of band structure during doping; (c) both CB shift and B–M shift of band structure when the carrier concentration $C < C_{Mott}$; (d) CB shift, B–M shift, and shrinkage effect of band structure when the carrier concentration $C > C_{Mott}$.

The influence of the B element on the E_g is also studied. The calculated E_g of pure and doped ZnO nanoparticles are presented in Figure 7. Different from Mg-doped ZnO nanoparticles, the E_g of B–Mg co-doped ZnO increases first and then decreases while the doping amount of Mg element gradually increases. A small number of carriers induced by

doping will cause Burstein–Moss shift, broadening E_g , as shown in Figure 6c. However, when the concentration of carriers is higher than the critical concentration of Mott transition (C_{Mott}), Urbach tail will cause the shrinkage of energy gap [29], which is shown in Figure 6d. It is known that the replacement of B^{3+} atom with host Zn^{2+} atom generates a large amount of carrier, and excessive carrier concentration results in the decreasing of E_g (Shrinkage effect).

Figure 7. Band gap energy E_g of pure and doped ZnO nanoparticles. (a) $Zn_{1-x}Mg_xO$; (b) $Zn_{1-0.02-x}Mg_xB_{0.02}O$; (c) $Zn_{1-0.04-x}Mg_xB_{0.04}O$.

4. Conclusions

The pure and doped ZnO nanoparticles were successfully synthesized by a simple one-pot method. XRD results show that both Mg and B are incorporated into the ZnO lattice without changing the original wurtzite structure of ZnO. By introducing Mg element into ZnO nanoparticles, the lattice parameter is increased. However, the B element increases the lattice strain and dislocation density, thereby decreasing the crystallite size. Combined with the analysis of the microstructure, it can be seen that Mg ions promote the crystallization of ZnO, while B ions make the crystallinity worse. The influence of doping Mg and B on the band structure is also investigated. The results show that the E_g of ZnO nanoparticles increase with doping Mg, while that of B-Mg co-doped ZnO increases first and then decreases due to the competition of CB shift, Burstein–Moss shift and Shrinkage effect.

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