Effects of (Ce, Cu) Co-doping on the Structural and Optical Properties of ZnO Aerogels Synthesized in Supercritical Ethanol

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Abstract. Undoped, Ce-doped, Cu-doped and (Ce,Cu ) co-doped ZnO aerogels were synthesized by sol-gel process in supercritical conditions of ethanol. [Cu]/[Zn] and [Ce]/[Zn] atomic ratios were fixed at 0.02 (2%). The aerogels were investigated without any additional treatments by using X-ray diffraction (XRD), UV–visible spectrophotometry, scanning electron microscopy (SEM), Energy-dispersive X-ray spectroscopy (EDS), Fourier transforms infrared spectroscopy (FTIR) and photoluminescence spectroscopy (PL). XRD results revealed that all the samples are well crystallized in hexagonal wurtzite structure. EDS measurements showed that highly pure aerogels are prepared. SEM analysis indicated that the morphology of the samples is dependent on Cu and Ce dopants. From UV-visible spectroscopy analyses, it was shown that the absorption and the band gap of the aerogels are strongly affected by Ce and Cu dopants. FTIR spectra demonstrated that co-doping induces a shift of Zn-O bond vibration band toward low wavenumbers. The room temperature photoluminescence spectra put into evidence that the visible emission intensity is influenced by Ce and Cu doping. In particular, the co-doping leads to the appearance of a blue emission band at 443 nm.

1. Introduction

Zinc oxide (ZnO) has been extremely studied as a multifunctional material for spintronics, optoelectronics, catalytic and photovoltaics applications [1-6]. Doping is a well known method to improve the physical properties of large gap materials. The introduction of extrinsic elements frequently affects the structural and optical properties of the material, but it is difficult to prove if these changes are due to the dopant elements or to the variation of native defects concentration induced by doping. Because of its high optical transmittance in the visible region and its important electrical conductivity, zinc oxide is highly recommended for photovoltaic applications [7].

ZnO particles are generally doped with transition metals or rare earths atoms in order to exalt its physical properties. Cu-doped ZnO nanopowder has been extensively investigated for use in particular applications [8-10]. Furthermore, Ce-doped ZnO was also studied in order to alter its optical characteristics [11-13]. Assadi et al. [14] have reported the enhancement of ZnO optical and magnetic properties by co-doping. Amiri et al. [15] have also demonstrated that (Ce, Cu) co-doped ZnO creates donor impurity bands producing a super-exchange interaction between the two atoms. Moreover, it has been reported that the interaction between Ce and Cu ions results in synergetic effects involving an easy creation of surface oxygen vacancies, leading to an improvement in catalytic activity [16, 17]. Zhang et al. [18] also reported the improvement of the optical activity of ZnO by Ce doping.
(Ce, Cu) co-doped ZnO nanoparticles have been synthesized with different processes such as solution combustion [19], microwave combustion [16], microemulsion reaction in mild conditions [20]. Up to now and to the best of our knowledge, (Ce, Cu) co-doped ZnO aerogels synthesized in superfluids has not been yet reported. In this context, the present work aims to complete our previously reported studies [21-23]. The choice of the sol-gel process followed by drying in supercritical conditions of organic fluids as synthesis route is justified by its simplicity, non-toxic chemicals, and low cost and large-scale nanostructures productions.

In this paper, undoped, Cu-doped, and (Ce, Cu) co-doped ZnO aerogels have been successfully synthesized via sol-gel method in supercritical drying conditions of ethanol. Structural, morphological and optical properties of the prepared samples have been investigated.

2. Experimental procedures

Undoped zinc oxide aerogel was synthesized by dissolving 16 g of zinc acetate dihydrate \([	ext{Zn} (\text{COOCH}_3)_2, \text{2H}_2\text{O}]: 95\%, \text{Aldrich}\) in 112 mL of methanol under constant magnetic stirring for 15 min. Copper acetate monohydrate \([\text{(CH}_3\text{COO})_2\text{Cu, H}_2\text{O}), \text{Proanalysis, 99\%}\], cerium nitrate \([\text{CeNO}_3,6\text{H}_2\text{O})\] were added to the mixture in order to get Cu-doped, Ce-doped and (Ce, Cu) co-doped ZnO aerogels. The atomic dopants concentrations were fixed at 2 % in all the sols \([\text{Cu}/[\text{Zn}] = 0.02, \text{[Ce]/[Zn]} = 0.02 \text{ and [Ce]/[Zn]} = 0.02\)). After complete dissociation of these sources, each sol was poured into an autoclave (Parr 4848 Reactor) containing ethanol. Then, the mixture underwent a drying at supercritical conditions of ethanol \((T_c = 243 \degree C, P_c = 6.3 \times 10^6 \text{ Pa})\). Once the supercritical conditions were reached, the solvent was removed and the autoclave was allowed to cool spontaneously to room temperature. The obtained aerogel was characterized without any chemical or heating treatments.

The crystalline structure of the as-prepared aerogels was studied using a PanAlytical diffractometer where the X-rays are produced from a radiation source CuKα \((λ = 0.154 \text{ nm})\), with an acceleration voltage of 40 kV and a current of 30 mA. Infrared spectra (FT-IR) were recorded with a Shimadzu’s IRAffinity-1 spectrometer by KBr pellet technique. SEM micrographs were recorded by a Quanta 2000 microscope with energy dispersive X-ray (EDS) spectroscope. EDS was used to identify the composition of the obtained products. UV–visible absorption spectra were recorded at room temperature using a Safas UVmc2 spectrophotometer. The photoluminescence properties were measured at room temperature using Xenon lamp operating at 350 nm. The incident beam was focused perpendicularly to the samples and the fluorescence was collected in a guiding mode using a TRIAX 550 Horiba-Jobin Yvon monochromator equipped with a nitrogen cooled CCD camera.

3. Results and discussion

XRD patterns of undoped, Cu-doped, Ce-doped and (Ce, Cu) co-doped ZnO aerogel powders are shown in figure 1. All the peaks can be indexed to ZnO hexagonal wurtzite structure (JCPDS 36-1451). As can be seen, no diffraction peaks of Ce, Cu or their related oxides are detected indicating the introduction of the dopants in the ZnO crystal structure. XRD diffraction peaks have been found to be quite sharp and intense which implies high crystallinity of the prepared aerogels. It can also be observed that doping ZnO with Cu atoms leads to the increase in XRD peaks intensities indicating the enhancement of the aerogel crystalline quality. This result implies, in the one hand, the occupation of \(\text{Zn}^2+(0.074 \text{ nm})\) vacancies by \(\text{Cu}^{2+}\) ions \((0.072 \text{ nm})\) [24]. In the other hand, the strong covalency of Cu-O makes the interaction of Cu with O stronger than that of Zn with O leading to the decrease of the oxygen defects in the lattice, which promotes the crystallinity of the aerogels nanoparticles [25].
Figure 1. XRD spectra of pure, doped and codoped ZnO aerogels

Figure 2. Most pronounced XRD peaks of the aerogels

The XRD spectra of the most pronounced peaks of figure 1, from which the principal structural properties of ZnO aerogels were determined, are shown in figure 2. Moreover, as can be seen from figure 2, the Ce doping induces the broadness and the shift to the lower diffraction angles without affecting the intensities of the XRD peaks of ZnO aerogel. This result indicates the increase of both the ZnO lattice parameters and the decrease in the crystallite size with Ce-doping. Furthermore, the introduction of Ce$^{3+}$ and Cu$^{2+}$ ions simultaneously in the lattice of ZnO deteriorates the crystalline quality of the aerogel. It was already reported that the intensity of the diffraction peaks decreases after the introduction of Ce and Cu atoms as compared to that of undoped ZnO due to the random occupation of the intrinsic defects in ZnO lattice by dopant ions and the difference in the ionic radii of zinc, cerium and copper [16,26].

ZnO lattice parameters $a (a = \lambda/(3^{1/2} \sin \theta))$ and $c (c = \lambda/\sin \theta)$ were calculated from the positions of the (100) and (002) peaks respectively. The degree of crystallinity $X_c$ is calculated using the following empirical relation [27]:

$$X_c = (0.24/\beta_{002})^3$$

(1)

where $\beta_{002}$ is the full width at half maximum (in degrees) of (002) Miller’s plane. The crystallites size and lattice micro-stress have been calculated using Williamson –Hall (W-H) method. In the case when both size and lattice micro-stress effects on the XRD line broadening are operative, Williamson and Hall have suggested a method combining the two effects to calculate these parameters. The W–H equation is expressed in the form [28]:

$$\beta \cos \theta = \frac{0.89 \lambda}{D} + 4\varepsilon \sin \theta$$

(2)

Where $\varepsilon$ is the stress associated with the nanoparticle, $D$ is the crystallite size, $\lambda$ is the X-ray wavelength (1.5418Å), $\beta$ is the full width at the half-maximum of the XRD line (in radians) and $\theta$ is the diffraction angle. The stress and the particle size can be obtained by plotting $(\beta \cos \theta)$ versus $(4 \sin \theta)$ for the 6 most important DRX peaks as shown in figure 3. The slope of the line $(4\varepsilon)$gives the stress and the intercept $(0.89\lambda/D)$of this line on y-axis gives the particle size$(D)$. The cell parameters
and $c$, average crystallites size $D$, Zn-O bound length ($L$), microstrain ($\varepsilon$) and the degree of crystallinity are calculated for all the aerogels and are shown in Table 1.

![Figure 3. W-H analysis of the pure, Cu-doped, Ce-doped and codoped ZnO aerogels.](image1)

![Figure 4. FTIR curves of pure and codoped ZnO aerogels.](image2)

**Table 1.** Cell parameters $a$ and $c$, average crystallites size $D$, Zn-O bound length ($L$), micro-strain ($\varepsilon$) and the degree of crystallinity $X_c$.

| Sample                  | $a$ (Å) | $c$ (Å) | $L$ (Å) | $D$ (nm) | Micro-strain ($\varepsilon$) | $X_c$ |
|-------------------------|---------|---------|---------|----------|-----------------------------|-------|
| Undoped ZnO             | 3.2451  | 5.1996  | 1.9749  | 53       | $3.6 \times 10^{-4}$        | 1.094 |
| Cu-doped ZnO            | 3.2506  | 5.2072  | 1.9781  | 62       | $9.7 \times 10^{-4}$        | 1.375 |
| Ce-doped ZnO            | 3.2509  | 5.2076  | 1.9783  | 37       | $7.6 \times 10^{-4}$        | 0.555 |
| (Ce,Cu)codoped ZnO      | 3.2606  | 5.2221  | 1.9841  | 29       | $7.9 \times 10^{-4}$        | 0.281 |

The average crystallites size of the particles has been found to be 53, 62, 37 and 29 nm in undoped, Cu-doped, Ce-doped and (Cu,Ce)-codoped ZnO aerogels, respectively. This results indicates that the simultaneously presence of Cu and Ce atoms in the aerogel leads to the diminution of the particles size due probably to the decrease of molecular concentration at the crystallites surface of the aerogel [29]. Also, It has been reported that smaller size of the codoped samples is caused by the formation of Zn–Ce–Cu–O in the crystal lattice, which plays an important role in hindering the crystal growth [30]. The best crystallinity has been found in the Cu-doped ZnO aerogel and the worst one has been found in codoped aerogel. This result confirms the XRD measurements. For Cu-doped ZnO aerogel, the dilation of the cell parameters can be due to the tensile stress exerted by the solvant during the supercritical drying process on the Cu-O bond. Kuluk et al. [31] reported that for low Cu
concentration, the substitution of Cu$^{2+}$ ions instead of Zn$^{2+}$ ions at their lattice sites increases the lattice constants and the interplanar distance, which would lead to the decrease of the diffraction angle compared with undoped ZnO. The increase of the cell parameters in Ce-doped aerogel can be explained by the difference in ionic radii of Zn$^{2+}$(0.074 nm) and Ce$^{3+}$(0.114 nm) when Ce ions occupy Zn$^{2+}$ sites in ZnO lattice. The results show that in the codoped ZnO aerogels Ce and Cu ions can simultaneously substitute Zn vacancies in the same cell and the tensile strain exerted by the solvent during the drying process can participate to the increase of Zn-O length bond. This suggestion can be explained by the value of microstrain obtained in ZnO aerogel in which Ce and Cu atoms are simultaneously introduced (Table1). In the other hand, the incorporation of cerium ions increases the lattice parameters in (Ce, Cu) codoped ZnO compared to Ce-doped ZnO aerogel. This indicates that Ce$^{4+}$(0.101 nm) and Ce$^{3+}$(0.114 nm) ions occupy Zn$^{2+}$(0.074 nm) sites in Ce-doped ZnO and (Ce, Cu) codoped ZnO, respectively. The presence of Cu$^{2+}$ ions in the codoped aerogel favors the replacement of Zn$^{2+}$sites by Ce$^{3+}$ thus dilating the cell parameters of the ZnO lattice. Again, ZnO resultant crystalline structure was the hexagonal wurtzite structure for all the synthesized ZnO aerogels. The recorded FT-IR spectra of pure, Ce-doped, Cu-doped and (Cu, Ce) codoped ZnO aerogels are shown in figure 4. The vibrational bands with different intensities are observed in all spectra approximately at the same positions (3482, 2366, 1586, 1417 and 1036 cm$^{-1}$). No bonds related to the dopants or dopant-based compounds are found in the FT-IR curves. The small intensities of the lines testify the high chemical purity of the elaborated aerogels. Compared to those of the undoped aerogel, the bands located at 1586, 1417 and 1036 cm$^{-1}$ are amplified in Ce-doped and (Cu, Ce) codoped ZnO aerogels. In Cu-doped aerogel the intensity of these bands is diminished. The absorption band at 3482 cm$^{-1}$ is due to the stretching mode of O-H group which reveals the existence of small amount of water absorbed by the ZnO aerogels. The peak at 1036 cm$^{-1}$can be attributed to aromatic C=C stretching mode [31]. The absorption band at 1586 cm$^{-1}$ results from the C=O asymmetric stretching vibration of zinc monoacetate [33]. The band located at 1417 cm$^{-1}$ is assigned to C-C stretching bond peak [35]. A very strong band between 400 and 460 cm$^{-1}$ due to the vibration of the Zn-O bond characterizes the spectra. This band is observed at 453, 436 and 428 cm$^{-1}$ in Ce-doped ZnO, Cu-doped ZnO and (Cu, Ce) codoped ZnO aerogels, respectively. The presence of Ce atoms in the aerogels leads to the intensification of Zn-O bond. The simultaneously presence of Ce and Cu ions in ZnO lattice induces the shift of Zn-O bond vibration band toward low wavenumbers.

Figure 5 shows SEM images and EDS result of Ce-doped, Cu-doped and (Cu, Ce)-codoped ZnO aerogels. The Ce-doped ZnO particles agglomerate to form torus-like shape grains with a high density. The image of Cu-doped ZnO aerogel shows homogeneous quasi-spherical-like nanostructures. The particles are less agglomerated and appear as “snow”or a “frozen fume”. In (Ce, Cu)-co-doped aerogel SEM micrograph, we can observe the presence simultaneously of the two previous morphologies (torus-like and spherical-like). The changes in the grains morphologies could be related to the nucleation mechanism in the growth process of the particles. This is because a restrained nucleation rate and a subsequent lower growth rate of the aerogels may appear when the ionic radii of Ce$^{3+}$ and Cu$^{2+}$ ions are different from that of Zn$^{2+}$[16].

The EDS data of these aerogels show the presence only O, Zn, Cu and C atoms. The C atoms come from the grid used in the experimentation. Without taking into a count C atomic percentage, [Cu]/[Zn] atomic ratio has been found to be 2.7 % and 3.1 % in Cu-doped and (Ce, Cu)-codoped ZnO aerogels, respectively. However, [Ce]/[Zn] has been found to be 3.4 % and 2.3 % in Ce-doped and (Ce,Cu)-codoped ZnO aerogels, respectively. This result indicates that the percentage of Ce, Cu and Zn values obtained did not strongly deviate from their initial stoichiometry.
The normalized optical absorption spectra of all elaborated aerogels are shown in figure 6. The absorption edge is found to be at 346 nm (3.58 eV) in the undoped ZnO, 350 nm (3.54 eV) in Ce-doped ZnO, and 353 nm (3.51 eV) in Cu-doped ZnO and 352 nm (3.52 eV) in (Ce, Cu)-codoped ZnO aerogels. After the introduction of dopant elements in the ZnO lattice, the absorption edge shifts to higher wavelengths. This shift is probably due to the formation by the dopant elements of an impurity band, which overlaps with the conduction band edge and leads to the decrease of the optical gap. Muthukumar et al. [36] reported the decreasing in the bandgap of Cu doped ZnO nanoparticles synthesized by co-precipitation method for low Cu concentration. Panda et al. [2] reported that the UV band observed at 368 nm in pure ZnO is shifted to 372 nm in cerium (1.5 %) doped ZnO synthesized through soft solution route. This band is attributed to the band edge absorption of ZnO and this band is compressed by cerium doping. Djadja et al. [37] reported the same behavior of the absorption edge in Ce-doped ZnO nanoparticles prepared by the co-precipitation method. They believed that the incorporated Ce ions into ZnO matrix lead to the formation of electron localized states into the bandgap of ZnO. These electron states locate closer to the lower edge of conduction band to form the new lowest unoccupied molecular orbital, which results in the band gap reduction. In (Ce, Cu)-codoped ZnO spectrum a shoulder at 340 nm (3.64 eV) is observed. Based on the first principle theoretical calculations carried out to study the effect of Ce and Cu dopants on the structural stability and magnetic properties of ZnO, Arul Mary et al. [16] found the formation of two new bands in both the valence and conduction bands near the Fermi energy. They suggested that the generation of these bands is due to the interactions between Cu (d), O (p) and Ce (f) electrons. Based on these data, the
decrease of the optical gap can be explained by the formation of impurity bands at the bottom of the conduction band and/or at the top of the valence band.

The room temperature photoluminescence spectra of the undoped, Cu-doped, Ce-doped and (Ce, Cu) co-doped ZnO aerogels are shown in figure 7. As can be seen, the PL spectra of the undoped and Cu-doped ZnO aerogels consist of a sharp UV emission and a broad visible one. However, Ce doping and codoping leads to the quenching of the broad visible emission and the appearance of two sharp emissions centered respectively at 546 and 612 nm. In particular, Ce co-doping aerogel leads the emergence of a relatively weak and broad blue emission at 443 nm. The narrow UV emission observed at 386 nm is due to near-band edge emission of the ZnO. The visible emission observed in the PL spectra of the undoped and Cu-doped ZnO demonstrates that this emission consists of 3 bands centered at 510, 546 and 617 nm.

![Figure 6. Absorption spectra of undoped, doped and codoped ZnO aerogels](image6.png)

![Figure 7. RT PL spectra of undoped, doped and codoped ZnO aerogels](image7.png)

The green emission at 510 nm is assigned to the transition related with the single ionized oxygen vacancies [37]. Many authors attribute this emission to the transitions between the singly ionized oxygen vacancies to the photoexcited holes [38]. The green emission at 546 nm observed in all the aerogels comes from the recombination of electrons in singly occupied oxygen vacancies with photoexcited holes [39]. The orange emission observed at 617 nm (2.03 eV) is usually attributed to oxygen interstitials [40].

It is remarkable that the introduction of cerium in the ZnO (Ce-doped and (Ce,Cu) codoped samples) reduces considerably the intensity of the visible emission. In particular, the green (510 nm) emission is totally quenched. The reduction in total emission upon Ce doping can be attributed to the increase in non-radiative transitions. This quenching may be due to the introduction of Ce atoms in Vzn sites. It is noted that several reports have attributed the red emission at 2 eV to Vzn [41,42]. Furthermore, the decrease of the visible emission intensity after the introduction of Ce atoms can be assigned to the increase of non-radiative relaxation, which is due to the surface bound states introduced by cerium ions and the degradation of ZnO crystal quality [43,44]. In the PL spectrum of (Ce, Cu) co-doped ZnO aerogel, a new emission band is observed at 443 nm. It has been reported that this emission originates from the electron transition from shallow donor levels of oxygen vacancies to the top of valence band levels [45]. Panda et al. [12] reported a visible light absorption band at 438 nm in cerium doped ZnO synthesized via soft solution route. They attributed this emission to cerium ions. The appearance of this emission can be explained in the light of Ce$^{3+}$ (4f$^5$5d$^1$6s$^1$) incorporation into ZnO there by forming empty energy levels below the conduction band [12]. In addition, to this emission can contribute the transitions between impurity energy levels
and conduction band of ZnO or 4f–4f or 5d–5d transitions in Ce$^{4+}$ states. One of the explanations for the appearance of Ce$^{3+}$ emission in the co-doped aerogel resides in the difference of the ions radii. As Ce$^{3+}$ ions are bigger than Zn$^{2+}$ ions, the incorporation of trivalent Ce$^{3+}$ into ZnO could cause a big distortion in the ZnO crystal lattice and also require association with other charge-compensating lattice defects or impurities, such as Cu$^{2+}$ ions [46]. The appearance of this emission only with the presence of Cu atoms can be explained by the fact that Cu$^{2+}$ ions might change the Ce$^{3+}$ ions environment and increase the transition probabilities with the result of inducing the energy transfer from ZnO to Ce$^{3+}$ ions [47,48].

4. Conclusion

Undoped, Ce-doped, Cu-doped and (Ce, Cu)-codoped ZnO aerogels were successfully synthesized in supercritical ethanol. [Cu]/[Zn] and [Ce]/[Zn] atomic ratios were fixed to 0.02 (2%) in all solutions. The (Ce, Cu) iso-codoping caused the changes in the crystallite size, lattice parameters and strain of ZnO lattice. XRD characterizations manifest that the aerogels have high crystallinity and shared the presence of wurtzite structure. (Ce, Cu)-codoping strongly deteriorates the crystalline quality of the ZnO aerogel. The presence of Cu$^{2+}$ ions in the codoped aerogel favors the replacement of Zn$^{2+}$ sites by Ce$^{3+}$ thus dilating the cell parameters of the ZnO lattice. EDAX studies confirm the high purity of the aerogels and the incorporation of dopant atoms in ZnO lattice. FT-IR spectra confirm that the presence of Ce and Cu ions simultaneously in ZnO lattice induces a shift of Zn-O bond vibration band toward low wavenumbers. The introduction of dopant elements in the ZnO lattice shifts the absorption edge toward higher wavelengths implying a decrease in the optical band gap. The room temperature photoluminescence spectra show a narrow UV emission at 386 nm and a broad visible luminescence edge toward higher wavelengths implying a decrease in the optical band gap. The room temperature photoluminescence spectra show a narrow UV emission at 386 nm and a broad visible luminescence edge toward higher wavelengths implying a decrease in the optical band gap. The room temperature photoluminescence spectra show a narrow UV emission at 386 nm and a broad visible luminescence edge toward higher wavelengths implying a decrease in the optical band gap. The room temperature photoluminescence spectra show a narrow UV emission at 386 nm and a broad visible luminescence edge toward higher wavelengths implying a decrease in the optical band gap. The room temperature photoluminescence spectra show a narrow UV emission at 386 nm and a broad visible luminescence edge toward higher wavelengths implying a decrease in the optical band gap.

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