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A Review On Optical Properties Of Transition Metal Doped Zno Nanostructures

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***Abstract:** Nanostructures of ZnO have received considerable attention due to their wide range of applications in solar cell, photovoltaic devices, transducers and optoelectronics. This work represents a comparative study on optical properties of transition metal doped ZnO nanostructures. There are different methods for the growth of ZnO nanostructures and their doping processes. This work reviews different growth mechanism and variation of optical properties of ZnO nanostructures because of metal doping. A strong correlation of changes in light absorption, photoluminescence, energy band gap and lattice parameters are well explained with reference to doping of Cu and Co transition metals.*

***Keywords:** Growth Mechanism, Kinetics of Nanostructures, Optical Properties.*

I. INTRODUCTION

The fascinating and surprising optical properties of ZnO nanostructures have attracted much attention for application in optoelectronics, photonics, sensors, photovoltaic devices as well as in the field of life sciences. Zinc oxide is a unique material with direct energy band gap 3.37 eV. These are un-expensive material having optical transparency in visible light region. In absence of doping, most of these structures are found as n-type material. The oxygen vacancies and zinc interstitials may be responsible for this n-type behavior. Again extra substitution of hydrogen impurities are also considered as one of the cause of this behavior [1-3].

The energy band of ZnO makes it suitable for short wavelength range optoelectronic device fabrication. The conductivity of ZnO can be enhanced by the method of doping. Again these nanostructures can be found as a diverse group of growth morphologies. These are available as nanopins, nanobelts, nanowires and nanorods in one dimensional structure. These nano structures are again found in 2D structures as nanosheet, nanoplates and nanopellets. On the other hand, 3D structures of zinc oxides are obtained as flowers, snowflakes and dandelion etc. Zinc oxide is II-VI semiconductor element with exciton energy of 60 mV. The zinc oxide crystallizes are available in three forms as hexagonal wurtzite, cubic zincblende and rarely observed as cubic rocksalts. The common stable form at ambient condition is wurtzite structure. The large direct band gap of ZnO associated with higher breakdown voltage, sustainability for large electric field and higher temperature operation for device fabrication realization. Zinc oxide is known as multifunctional material because of its chemical stability, high electrochemical coupling coefficient, high photostability and broad range of radiation absorption [4-8].

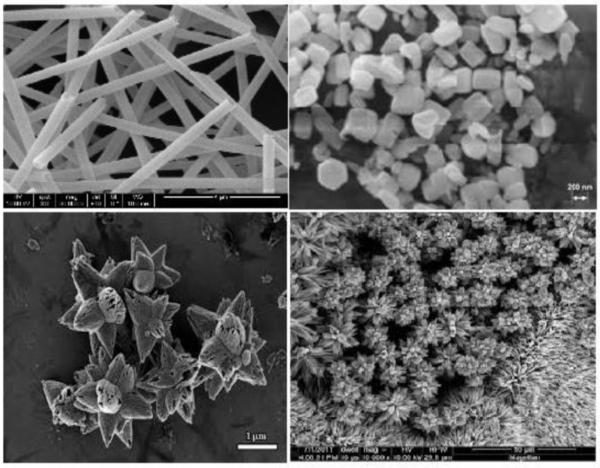

Figure 1: Different structures of ZnO nanostructures

The reductions in size of zinc oxide to nanometer range caused quantum confinement effect and novel optical, electrical properties of ZnO Nanoparticles were observed by different workers. One dimensional ZnO nanowires were reported as ideal system for study of transport process in low dimension systems for development of new generation nano-devices with higher performance. In wurtzite structure of zinc oxide material, the piezoelectric and pyroelectric properties were utilized for sensor fabrication. The lacks of centre of symmetry along with large electrochemical coupling properties of these structures were made them potential candidates for sensor expansion [9-11].

The wurtzite structure of ZnO is composed of alternating planes arranged with tetrahedrally coordinated with O^{2-} and Zn^{2+} ion which are alternately attached along the C-axis as shown in the Fig-1. This tetrahedral coordination is responsible for non central symmetric structure of ZnO. The surfaces of these materials are polar in nature and most common polar surface is the basal plane. Generally polar surfaces exhibit massive surface reconstruction to maintain a stable structure. The remarkable property of zinc oxide is that their surfaces are flat, stable and without reconstruction [12-15].

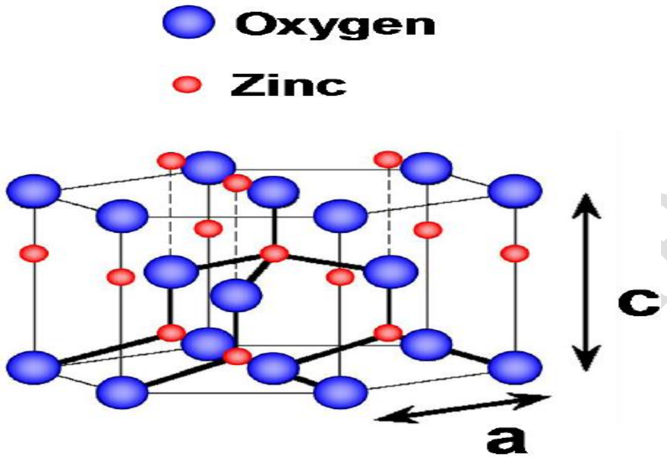

Figure 2: Wurtzite Structure of ZnO Nanostructures

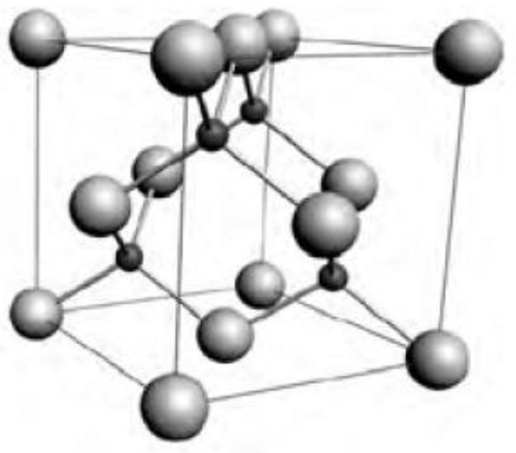

Figure 3: Zinc Blende Structure of ZnO Nanostructures (O atoms are white and Zn atoms are in black color)

The surface defects are responsible for the photo catalytic actives of metal oxides because the number of active sites can be enhanced by these defects. Due to this reason doping of transition metal are carried out in zinc oxide for variation of photo catalytic properties of ZnO nanostructures. The optoelectronic properties of ZnO have been used successfully for photonic device fabrication. The photoluminescence spectra of ZnO nanostructure have been reported for excitonic emission of light. The higher value of the exciton binding energies of ZnO nano particles were explained on the basis of quantum sized confinement effect. The strong peaks of ZnO nanostructures in luminescence spectra were observed due to band to band transition. The presence of oxygen vacancy caused the emission of green, yellow light. Again these green emission intensities of ZnO can be increased with decrease in particle size into nano meter range. The larger surface to volume ratio of nano particles lead to a higher level of defects and surface recombination which may be attributed to emission of spectra of ZnO nanostructure. The double ionized oxygen vacancies were responsible for the red luminescence of ZnO particles. The optical properties of ZnO confirmed it as a promising candidate for UV detector [16-18].

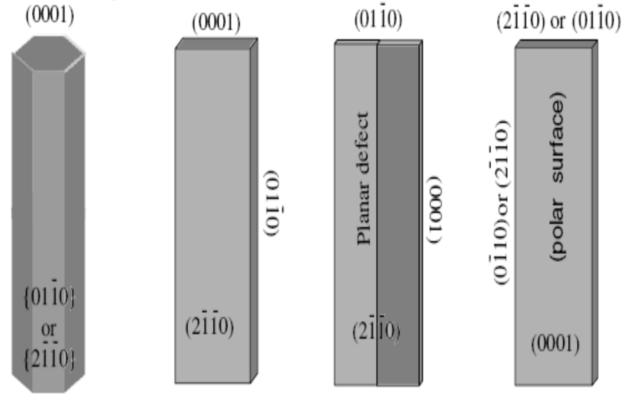

Figure 4: Different growth planes of ZnO nanostructures

II. GROWTH OF ZNO NANOSTRUCTURES

R.P. Singh et.al. have explained a simple method for synthesized of ZnO nano particles. Zinc Acetated dehydrates and sodium hydroxide were purchased from Merck and used as reagents. The co- precipitation method was adopted for production of ZnO nanostructure. An aqueous solution of Zinc Acetated dehydrates of 0.02 m was prepared in 50 ml of distilled water under continuous stirring. Then latex of Maddar (calotropis procera) was added after 10 minutes of stirring followed by addition of 2.0 m NaOH aqueous solution. The pH was maintained at 12 and the solutions were again stirred for another two hours. The precipitated were taken out and washed with distilled water and ethanol. White powders of ZnO nanostructures were obtained by drying the final product at 60^0 C in vacuum oven over night [19].

N. K. Hassan et.al. have used electro chemical deposition technique for growth of ZnO nano particles by using Zn $(NO_3)_2$ at room temperature. A simple two electron Teflon cell was used by them. A spiral platinum wire and Si (100) substrate were used as anode and cathode for electrolysis respectively. The distance between two electrodes was kept at 0.5 cm, the deposition time was 1.5 hours and the pH was maintained at 5. All products were annealed at 500^0 C under oxygen ambient [20].

A. Mansuri et.al. have shown the chemical growth of ZnO nanostructures by using Zn $(NO_3)_2$ $6H_2O$ (Zinc Nitrate Hexa hydrate) and $C_6H_{12}OH$ (Hexa Methylene tetramine) precursors under continuous stirring. The solution and cleaned microscope glasses were kept inside the auto cleavable bottle with polycarbonate sealed cap. The heating was carried out for 20 hours at 95^0 C for the growth process. Morphology of the prepared structure was controlled by adjusting pH of the solution by adding 0.1 M NaOH reagent. Finally the substrates were taken out from the solution and washed with distilled water. The substrate was dried at 95^0 C inside oven. The prepared ZnO nanostructures were characterized Bruker AXS D8 advanced X- ray diffraction analyzer (XRD, with Cu K_α radiation) and Cam Scan MV 2300 scanning electron microscope. In XRD analysis, they have found that the prepared ZnO nano wires were in single phase with a wurtzite hexagonal structure. They have also confined the formation of pure ZnO in absence of other peaks in the spectra. Again they have suggested that larger value of pH may be attributed to higher reaction rate, which result the poor crystal quality of the same samples. The morphology observed for ZnO nano wires at pH7. These were random in distribution with length of 2- 10 µm and of 150- 300 nm. In pH 12, they have flower like structure (ZnO nano particles) with length of 2-4 µm and diameter of 150- 500 nm. Therefore it is necessary to be mentioned here that the larger pH value may hamper the size of ZnO nano structures. In this work the diameter of ZnO nano flowers were found upto 500 nm. This work explained the formation of ZnO nanostructures initiative with thermal degradation of Henamethynetetramine (HMT) which was responsible for release of hydroxyl ions. The chemical reactions were summarized as given below

$6\,(CH_2)_6\,N_4 + 6H_2O \leftarrow\!\text{-}\!\rightarrow NH_3 + HCHO$ --- (1)

$NH_3 + H_2O \leftarrow\!\text{-}\!\rightarrow OH^- + NH_4^+$ ------ (2)

$H_2O + ZnO_{(s)} \rightarrow Zn\,(OH)_2 \leftarrow\!\text{-}\!\rightarrow Zn^2 + OH^{-2}$ ----- (3)

Simply, the growth kinetic of ZnO nano particles was shown dependence on the concentration of OH^- ions or the value of pH [21].

Zinc oxide nano particles were successfully prepared by wet chemical method by J. T. Ullakkya et.al. Zinc Acetate (purchased from Merck) and potassium hydroxide (Merck) were used as precursors where as ethanol was used as solvent for the growth of zinc oxide nano structures. Zinc acetate solution of 0.1 M molarity was prepared in ethanol with pH 5. The solution was stirred for two hours for the formation of homogeneous mixture zinc salt solution. Potassium hydroxide of 0.1 M was mixed in ethanol with pH of 11. They have added KOH solution drop wise in zinc acetate solution and stirred it at room temperature with pH7. The stirring was carried out for 3 hours by using magnetic stirrer. The nano structures were separated by filtration method and characterized for their optical properties. The grain sizes of the prepared samples were found as 52 nm calculated by using Scherrer's equation from XRD analysis data. The maximum transmittance value in the UV spectra was reported as 200 nm to 900 nm region in their experiment. The calculated value of energy band gap of prepared ZnO nano particles were found as 4.2 eV. Again the luminescence peaks of the prepared nano structures were found at 540 nm and 563 nm. The surface morphologies of ZnO nano particles were studied by SEM and TEM analysis. The spherical nano particles were reported with an average size of 33nm [22].

III. TRANSITION METAL DOPING IN ZNO

The chemical, optical, electrical and magnetic properties of ZnO nano structures can be easily tailored by doping of transition metal atom for device realization. The transition metal (TM) doped ZnO nano particles were extensively investigated due to low cost of synthesis and versatile application for optoelectronic device fabrication. The transition metal doped ZnO nano particles have received much attention as a potential candidate for solar energy conversion, transparent UV protection films, spintronic devices and chemical sensors [23-25].

S.B. Rana et.al. used (zinc nitrate) Zn (NO_3) and (Sodium Hydroxide) NaOH as precursors of ZnO particles. The synthesis of ZnO nano particles were carried out by addition of Zn $(NO_3)_2$ with NaOH in deionized water solution under constant stirring. Ethyl alcohol was used for removal of impurities by the method of washing. The final product was dried at 100 ^{0} C in an oven for 15 hours. They have also prepared doped samples of ZnO nano structure with Ca and Cu metal atoms. XRD (CuKα radiation, 40 KV, 30 mA) was employed for determination of phase purity of the prepared samples. They have reported XRD data by the Rietveld method using the program Rietca and extracted lattice constants were reported as given in Table – 1. The wurtzite type crystals were found in both pure and doped ZnO nano particles.

Sample	a(A^0)	C(A^0)	Particle Size (nm)
Commercial ZnO	3.2475	5.2023	28
ZnO annealed at 500^0C	3.2467	5.2007	34
ZnO annealed at 700^0C	3.2475	5.2013	40
$Zn_{0.99}Co_{0.01}O$ annealed at 500^0 C	3.2474	5.2012	29
$Zn_{0.995}Cu_{0.05}O$ annealed at 500^0 C	3.2663	5.2313	24

Table 1: Lattice constants from Rietveld Refinement and calculated particle sizes (Reconstructed from Reference 26)

No significant shift of lattice constants were observed for Co- doped ZnO particles. They have explained that it may be due to the quite similar values of ionic radii of Cobalt and Zinc atoms (i.e., Zn^{2+} = 0.60 Å, Co^{2+}= 0.58 Å). But the lattice constants were slightly increased for Cu doped ZnO nanostructures. In the above case, the radius of Co^{2+} (0.73 Å) was larger than that of Zn^{2+} (0.60 Å) in their tetrahedral coordination. The reduction in particle size was observed from XRD pattern for all doped samples. This may be caused by the reduction of sintering rate due to incorporation of dopant atoms into the ZnO lattice. They have reported that the grain sizes of the prepared nano structures were increased with increased with calcinations temperature. The grain size of ZnO nano particles were observed more than 100nm, calcinated at

700^0 c. Their EDS results shown that no other element but only Zn and O were present in the pure sample where as dopant along with oxygens were present in doped samples. This work has explained that the wurtzite crystal structure may be the most common structure for pure and Co, Cu doped ZnO nano structure. This PAC (Perturbed Angular Correlation) spectrum of this work also supported the formation of single phase ZnO. The lattice constants of ZnO were increased with Cu doping. Again the sizes of the ZnO nano particles were increased for higher calcinations temperature [26].

K. Mlenova et.al. have prepared copper doped ZnO nanoparticle by precipitation method. They have used $ZnSO_4.7H_2O$ (Zinc hydrate heptahydrate), sodium carbonate (Na_2CO_3) and $CuSO_4$. H_2O (Copper Sulfate) for synthesis of ZnO nanostructures. Sodium Carbonate (90gm) was dissolved in 850 ml of H_2O under heating and constant stirring for solution preparation. Another solution of $ZnSO_4$ $7H_2O$ (20gm) was prepared in 140 ml of H_2O. Doping of Cu- atom was carried out by different calculated quantities of copper sulfate to the $ZnSO_4.7H_2O$ solution. Again these mixture solutions were added to Na_2CO_3 solution drop wise and pH was maintained at 11. The final solution was stirred for 10 minutes. The precipitate was filtered and washed several times with distilled water. They have prepared three samples of ZnO doped with 0.24%; 0.35% and 1.07 of Cu metal atom. The Atomic Absorption Analysis (AAS) on FAAS- SOLAR M5 spectrometer was used for determination of chemical composition of the prepared samples. The XRD patterns were recorded by using TURM62 diffractometer with CoKα radiation. The formation of wurtzite phase of ZnO was confirmed from X- ray diffraction analysis. The optimal doping concentration Cu- metal atom was reported as 0.5% wt. They have found shrinking of unit cell of ZnO sample due to Cu doping. Similar results were also reported by Belini and Bahsi. Because of nearly equal atomic radii of Cu^{2+} (atomic radii 0.057 nm) and the Zn^{2+} (atomic radii 0.060 nm), during diffusion process, the Zn^{2+} cations were substituted by Cu^{2+} ions. They have also observed that Cu doping in ZnO samples resulted a little increased in the crystallite size than the un-doped samples. The X- ray photo electron spectroscopy (XPS) studies were performed by using AA VG Escalab II electron spectrometer (ALKα radiation, 14866 v energy) for investigation of surface composition and chemical state of Cu doped samples. They have calculated the segregation of Cu atom on the surface of ZnO Nanoparticles during doping process. They have well explained the photo catalytic activities of ZnO Nanoparticles by using TG- DTA. They have found that the pure Zn samples were with best photo catalytic properties. Similar results were also reported by other workers [27].

The effect of Co incorporation into ZnO nano articles was explained by N. F. Djaja et al. Zinc sulfate heptahydrate ($ZnSO_4$. $7H_2O$, 99% Marck) and cobalt chloride hexahydrate ($CoCl_2$. $6H_2O$) were prepared in distilled water. On the other hand a separate solution of NaOH was prepared in de- ionized water. The Co doped ZnO nano particles were prepared by adding both of the solution under constant stirring and pH was maintained at 12 by them. The X- ray spectroscopy (EDX) LEO 420 was employed for study of existence and composition of Co dopants in ZnO nano structures. The phase purity of the samples were studied by Philips PW 1710, Cu-Kα (a= 1.54060 A^0) in the range of 10^0 to 80^0. Fourier transform infra red (FTIR) was used for explaining the structural properties of the prepared samples. The optical properties of the samples were well explained by UV-Vis spectroscopy by using a Shimadzu UV-Vis Spectrophotometer with a spectral reflectance standard in the wave length range of 200- 800 nm.

Doping of Co- atoms in pure ZnO was confirmed by EDX analysis. The quantitative characterization of Co/ Zn ratio was found by calculating the area at the corresponding spectral k lines of EDX spectra. They have found the amount of Co doping in ZnO particles were in between 3% to 18%. The representative XRD patterns revealed the presence of wurtzite ZnO phase and this wurtzite phase was also found in all doped samples. Therefore the wurtzite structure of ZnO particle was not influenced by Co doping. They have suggested the Co occupancy in Zinc sites for doped samples. The preferential growth of (101) orientation along c- axis was observed for all samples. Gradual decreased lattice parameters were reported for doped Co atoms in ZnO lattice. This formation of wurtzite structures in Co- doped and undoped ZnO samples were also observed in FTIR measurements. The absorption peaks of FTIR spectra in the range of 400- 700 cm^{-1} were attributed to the ZnO stretching modes while peaks in the range of 1100 to 1600 cm^{-1} were corresponding to the Zn- OH bending mode. In UV spectroscopy measurement, the spectra of Co- doped ZnO particles have shown peaks at 468, 515, 577 and 629 nm. This observed peaks were clearly indicating that the presence of Co^{2+} ion with $3d^7$ high- spin configuration in a tetrahedral crystal field. They were presented as

$4A_2$ (F)--- → $^2A_1$ (G), $4A_2$ (F)---→ $^4T_1$ (P), and $4A_2$ (F)-- → $^2T_1$ (G) transitions in high spin states (S= 3/2).

These transitions confirmed that Co^{2+} ions substituted the Zn^{2+} in ZnO lattice. If the concentration of Co atom in ZnO was more than 6% then the peaks were smeared out and the intensity of the reflection spectra would be decreased. The energy band of all prepared samples were also calculated from the diffuse reflectance spectra by plotting the square of the kubelka- Munk function F $(R)^2$ versus the energy in electron volts. The linear part of the curve was used for calculation of direct band gap energy by extrapolating F $(R)^2$= 0 in the graph [28].

In situ doping of Cu into ZnO nano roads were carried out by using sol- gel approach by S. Y. Pung at al. The concentration of Cu doping in ZnO was possible because of high ionization energy of Cu and the low formation of energy of substitutional group1B elements. Nitrate tetrahydrate Zinc (0.02 M), mthenamne (0.02M) and cupric acetate nanohydrate (0.02 M) were used for production of Cu doped ZnO nano roads. The X- ray diffractometer (Bruker Advanced X- ray solution D8, CuKα radiation, =0.154 nm) was used for crystalline phase determination of the prepared samples and wurtzite phase of ZnO was continued. The diffraction peaks were identified for the (100), (002), (101), (102) and (110) planes of zinc oxide. The twin rods like structure of ZnO nano rods were observed by a field emission scanning electron microscope (Zeiss Supra 35 VP) attached with an energy dispersive spectroscope. This work explained that the incorporation of Cu^{2+} ions into ZnO made little effect on ‘the lattice parameters a and c of ZnO as the Cu^{2+} ions and Zn^{2+} ions have almost similar ionic radius. Further, particularly the c- axis lattice parameter was decreased from 5.1872 A^0 (undoped) to 5.1760 A^0 (20% mol % G) with higher Cu concentration on doping. In comparison to 'a'- lattice parameter, the accumulation effect of Cu^{2+} ions made reduction of 'C'- lattice parameter of ZnO nano rod. The (002) crystal plane of ZnO crystal was found to composed of either Zn^{2+} or O^{2-} ions. Their work also reflected that the length of Cu doped ZnO nano rods decreased with increase in Cu dopant concentration. The homogeneous nucleation was found to be more dominant than heterogeneous nucleation for the growth of Cu doped ZnO nano rods in that so- gel synthesis procedure. The edge of the UV – Vis absorption peak was utilized for calculation of optical band gap of Cu doped ZnO nano rods. With increase of Cu dopant concentration, the optical band gap of Cu doped ZnO nano rods were found to be decreased [29].

IV. CONCLUSION

This review work is focused on different growth processes as well as structural, optical characterization of transition metal doped zinc oxide nanostructures. The main text of the work is divided into growth procedures and characterization of ZnO nanoparticles. The most extensively explored growth mechanism approaches were discussed in this work. Chemical synthesis, Hydrothermal synthesis and Electrochemical synthesis are the commonly used methodology for production of undoped and doped ZnO nano structures. Different methods have been reported for doping of transition metal into ZnO nano structures. The optical and structural properties have been studied mostly at room temperatures. The structural characterizations of the prepared samples were done by XRD and SEM analysis by various workers. The hexagonal wurtzite phase of ZnO nanostructures were reported in all doped and undoped samples. A systematic study on optical properties of doping of transition metals Cu and Co were carried out in this work. The wurtzite phase of ZnO nanostructures were revealed even in the highly doped samples. Different morphologies of prepared samples were observed with respect to different synthesis procedures and different nature of dopant.

The hydrothermal synthesis of ZnO nano particle have advantages over other growth process because of use of simple equipments, low cost, uniform production and less hazardous procedures. The sizes of the nano particle can be controlled by adjusting reaction temperature, reaction time and concentration of precursors. The doping of transition metals (TM) have changed the morphology of the ZnO nanostructures. In Cu doped ZnO, the lattice constants were slightly increased. The reduction of particle sizes were also observed from XRD pattern for Cu doped samples by S. B. Rana et al. On the other hand, the sizes of the prepared nano structures were increased at higher calcinations temperature. Similar results were reported by K. Milenova et al. for Cu doped ZnO nanoparticles. The shrinking of unit cell of ZnO nano structure was observed in Cu doped samples. Zinc oxide nano structures doped with transition metals have aroused research interest due to surprising optical properties for optoelectronic device realization. In case of Co doped ZnO nanostructures, N. F. Djaja et al. have explained the occupancy of cobalt in zinc sites for all the samples. A regular decreased in lattice parameters were observed for Co doped ZnO lattice. The optical study was used for calculation of energy band gaps of the samples.

More efficient exciton emission of ZnO nano structures at room temperature were observed because of very large exciton binding energy of these materials. The existences of various transition metals in ZnO nanostructures become advantageous for optoelectronic and spintronic application. The exciton energy and radiative lifetime of colloidal zinc oxide are expected to be strongly affected by quantum confinement. The different surface to volume ratios of nanostructures with different diameters also affects the intensities of wave length emission and absorption by the TM doped nanostructures. The effect of different dopant, concentration of precursors and reaction temperature on the structure, grain size and energy band gap were discussed. The XRD analysis of all TM doped samples demonstrated that the nano structures were formed with wurtzite structure and the particle sizes were increased at higher temperatures. The different emission spectra may be observed due to oxygen vacancy or interstitial defects of transition metals. The results described in this work are useful for the development of optoelectronic devices fabricated from transition metal doped zinc oxide nano structures in near future.

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