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SOL-GEL SYNTHESIS AND CHARACTERIZATION OF FE- DOPED ZNO NANO POWDER

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ABSTRACT:

ZnO doping with transition metals like Fe, Co, Mn, Ni, Cr, or V, leads to materials with completely different behaviour towards magnetic and optical excitation. The precursor of $Zn_{(1-x)}Fe_{(x)}O$ with x = (0, 0.02, 0.04, 0.06, 0.08, 0.1) nano particles have been successfully synthesized by novel sol–gel method at 80°C. Band gap of $Zn_{(1-x)}Fe_{(x)}O$ samples have been evaluated using UV-Vis spectrometer. The powder samples have been characterized by X-ray diffraction (XRD), Fourier transform infrared spectra, scanning electron microscope (SEM) and energy dispersive analysis of X-rays (EDAX). The XRD patterns showed the formation of single phase and hexagonal wurtzite structure. The average particle size is found to be in the range of 20–40 nm. SEM results show the morphology of the particles is hexagonal structure. Energy dispersive X-ray analysis confirmed the presence of Iron, Zinc and Oxygen.

Keywords: *Zinc oxide, Iron, Sol-gel method, Nanopowder.*

1. INTRODUCTION

Materials for spintronics are receiving increasing attention in the last few years. A variety of materials, specially **diluted magnetic semiconductors**, has been investigated in this connection. The II–VI based dilute magnetic semiconductors (DMS) are very encouraging materials for spintronics applications as DMS show ferromagnetic nature at room temperature [1-4]. Extensive studies were made on transition metal (TM) doped II–VI compound semiconductors (such as Fe, Co, Ni, Mn etc.) [5-6].In recent years, transition-metal ions (Mn, Fe, Co and Ni) doped ZnO has greatly attracted the attention for their promising versatile application because of their specific optical, structural and magnetic properties [7-10]. Zinc

oxide (ZnO) is one of the most important multifunctional semiconductor materials for various applications due to the wide band gap (3.37eV) and high exciton binding energy (60Mev). ZnO is also for medical and environmental applications. Then, ZnO has potential commercially due to its low cost, non-toxic, abundant resources in the nature and environmental friendly. Therefore, in the present work, we report the effect of doping on the structural and optical and magnetic properties of ZnO nanopowder synthesized by sol-gel method at different doping concentrations Fe ions. It is observed that the optical band gap increase the doping concentrations of Fe ions.

2. Experimental

Powder Synthesis

The metal nitrates of Zinc and ferric were used as the raw materials. Ethylene glycol and oxalic acid were used as the chelating reagents. In a typical run, the appropriate amounts of Zinc nitrate [$Zn(NO_3)_2.6H_2O$], ferric nitrate [$Fe(NO_3)3.9H_2O$], were dissolved in ethanol to form solution and stirred continuously using a magnetic stirrer at 80° C temperature. Finally, gelling agents ethylene glycol and oxalic acid were added into this mixture. A light brown coloured sol -gel was obtained after stirring and hydrolyzing the mixed solution for 45 minutes. Then the gel is dried in hot air oven at 110°C. Subsequently, the dried precursor was calcined at 500°C for 3hrs complete crystallization and then milling process was carried out to get a nano powder.

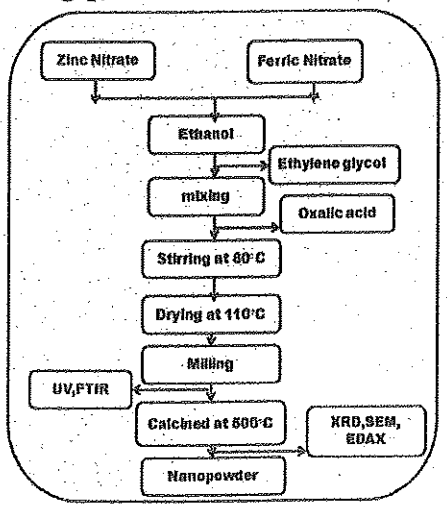

Fig.1 Flowchart for the preparation and characterization of $Zn_{(1-x)}Fe_{(x)}O$ nano- powders

3. Results and discussion

3.1 UV-VISIBLE SPECTROSCOPY

The optical band gap of the $Zn_{(1-x)}Fe_{(x)}O$ Powder from the reflectance spectra obtained by the UV–visible spectrometer in the range of 200 to 800 nm.. The bandgap increases from 3.33 eV to 3.57 eV with iron doping at different level percentage.

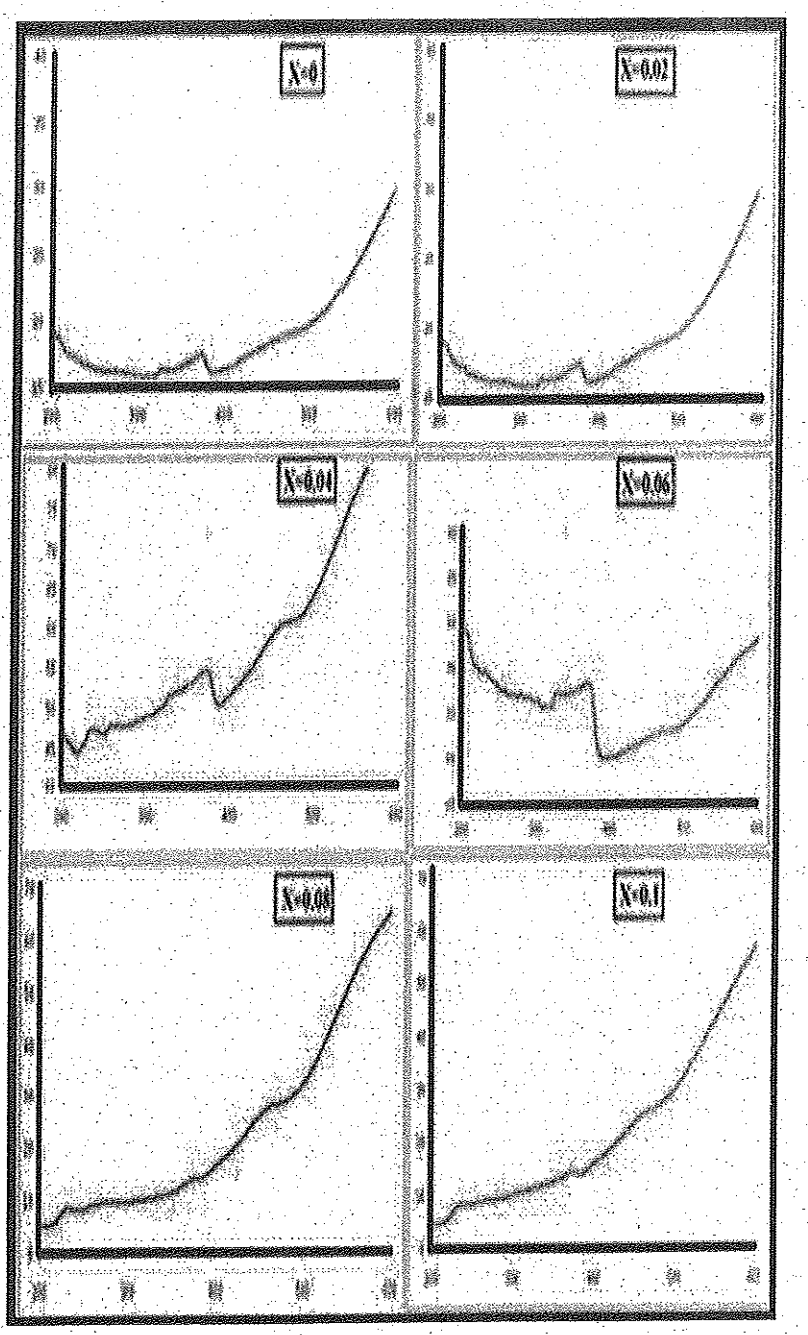

Fig.2: UV Reflectance of Fe doped ZnO Precursor

3.2 Fourier Transform Infrared Spectroscopy (FTIR)

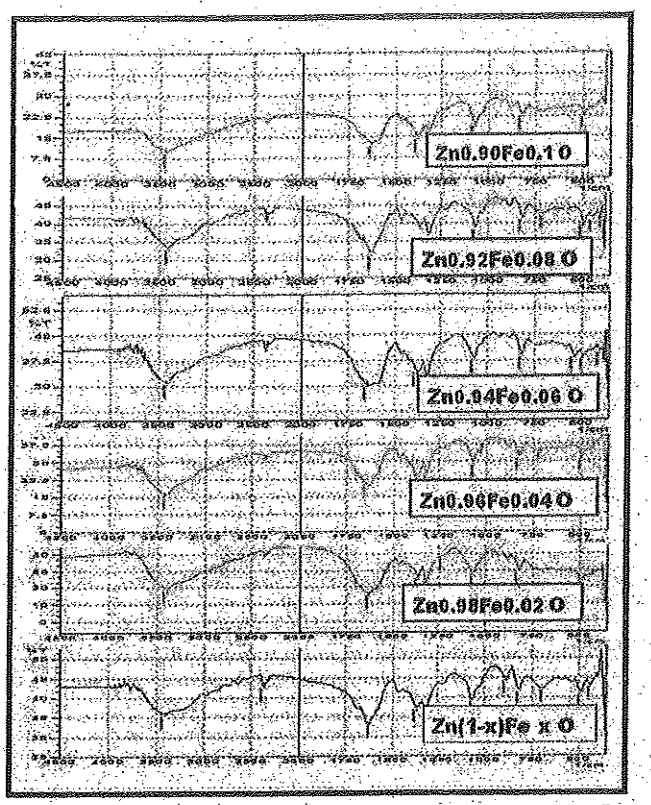

Fig: 3.FTIR Spectrum of Fe doped ZnO Precursor

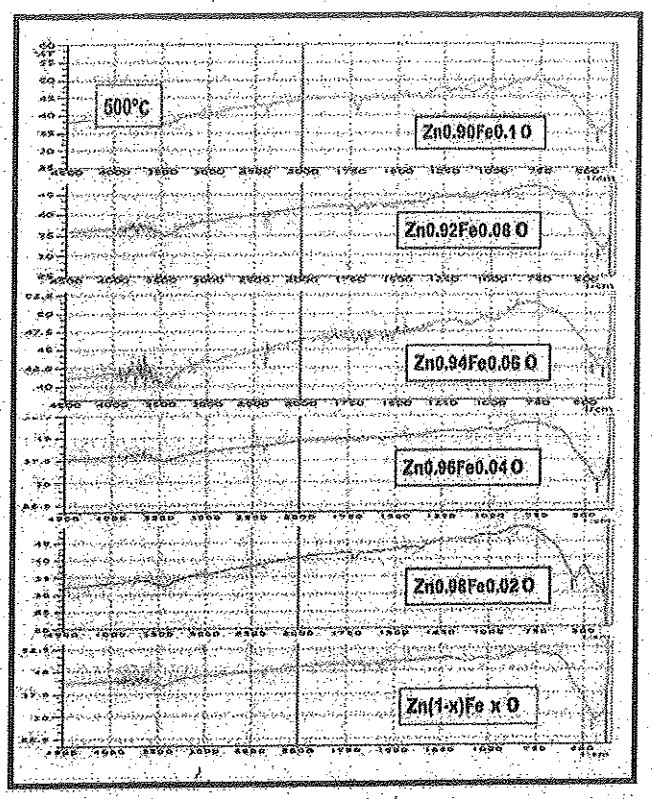

Fig: 4. FTIR Spectrum of Fe doped ZnO Calcined at 500°C

Figure 3 and 4 shows the FTIR spectra of the prepared samples. The reflectance band at 421–495 cm^{-1} in FTIR spectra is associated with the characteristic vibrational mode of Zn–O bonding.

3.3 X-RAY DIFFRACTION PATTEREN (XRD)

X-ray diffraction (XRD) is a powerful technique for determination of crystal structure and lattice parameters. Figure 5 shows the X-ray diffraction patterns of the $Zn_{(1-x)}Fe_{(x)}O$ compared to that of a pure ZnO. These patterns have been compared with standard JCPDS 89-1397.

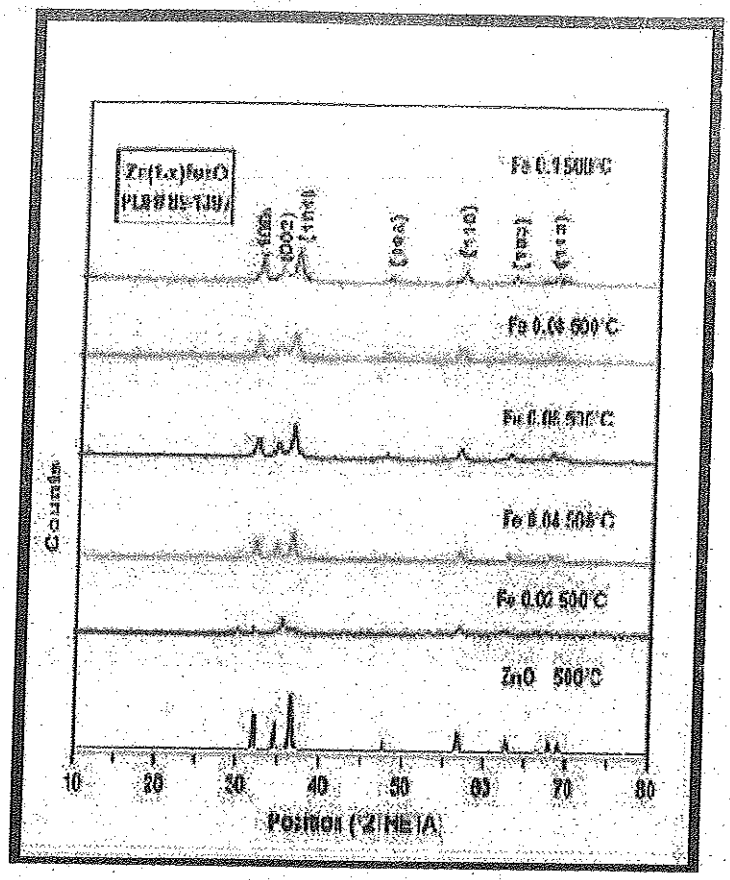

Fig: 5. X-raydiffraction patterns of the $Zn_{(1-x)}Fe_{(x)}O$ calcined in air at 500°C

The major planes corresponding to (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3) and (1 1 2) were found to be matched with wurtzite structure of ZnO. Figure 15 reveals that, there is no change in the wurtzite structure of ZnO after Fe doping. No extra peaks were found in the pattern indicating the formation of single phase $Zn_{(1-x)}Fe_{(x)}O$, This indicates that the doped Fe atoms substitute Zn atoms. Debye–Scherrer formula for finding the crystallite size (D) range is 20 - 40 nm.

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

Where, K is the Scherrer constant and has the value 0.9 for hexagonal crystal structures, λ is the wavelength of the radiation (1.5418 Å), b is the forward width at half maximum (FWHM) (radian) and h is the half value of the Bragg diffraction angle in the XRD pattern (degrees).

3.4 SEM (Scanning electron microscope)

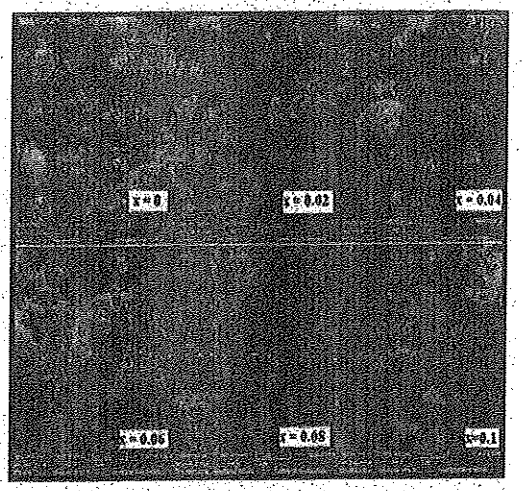

Fig: 6. SEM microstructures of the $Zn_{(1-x)}Fe_{(x)}O$, x = (0, 0.02, 0.04, 0.06, 0.08, 0.1) calcined in air at 500°C

Fig: 6 shows the SEM images of $Zn_{(1-x)}Fe_{(x)}O$, x = (0, 0.02, 0.04, 0.06, 0.08, 0.1) calcined in air at 500°C. It was observed that the morphology of the ZnO-NPs were hexagonal nanoparticles with diameter of about 20-50 nm. The average crystallite size of the Fe doped ZnO NPs decreased with increase in the Fe concentration from the different models implying that the inclusion of strain in different forms has very little effect on the average crystallite size.

3.5 ENERGY DISPERSIVE X-RAY (EDX) ANALYSIS

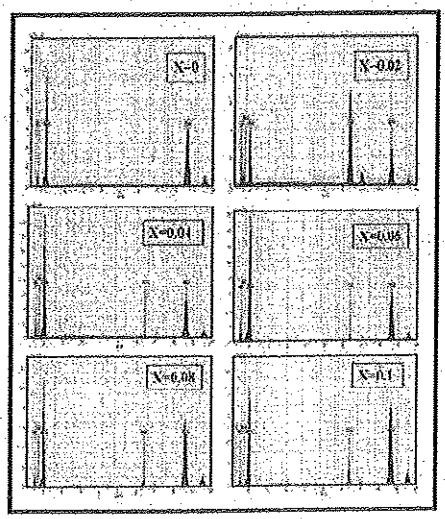

Fig: 7. EDX analysis of the $Zn_{(1-x)}Fe_{(x)}O$, x = (0, 0.02, 0.04, 0.06, 0.08, 0.1) calcined in air at 500°C.

The energy dispersive X-ray (EDX) analysis of $Zn_{(1-x)}Fe_{(x)}O$, x = (0, 0.02, 0.04, 0.06, 0.08, 0.1) calcined in air at 500°C was shown in Fig.7. Energy dispersive X-ray diffraction pattern shows the presence of Zinc, Iron and oxygen.

4. CONCLUSION

The precursor of $Zn_{(1-x)}Fe_{(x)}O$ with x = (0, 0.02, 0.04, 0.06, 0.08, 0.1) nano particles have been successfully synthesized by novel sol–gel method. The optical band gap of the $Zn_{(1-x)}Fe_{(x)}O$ Powder from the reflectance spectra obtained by the UV–visible spectrometer. The synthesized samples functional groups analyzed by Fourier transform infra-red (FTIR).The dried precursor powder was milled and then calcined at 500°C for 3 hrs. From X-ray diffraction, it was observed Fe-doped ZnO nanoparticles (NPs) have single phase and hexagonal wurtzite structure. The morphology and crystalline nature are characterized by Scanning electron microscopy (SEM), Energy dispersive X-Ray analysis (EDAX) confirmed the presence of Iron, Zinc and oxygen.

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