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Synthesis of Fe Doped Zno Spintronic Nano Powders by Solution Combustion Method & Their Characterization

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***Abstract*— In this work Fe doped ZnO powders were prepared by solution Combustion method using Zinc nitrate and ferric nitrate as oxidizer and urea as fuel at 500ºC. The synthesized powders were characterized by XRD for phase analysis, SEM for microstructure and EDS for elemental analysis. The obtained XRD patterns of synthesized powder were identical with the standard peaks of ZnO (JCPDS card number 36-1451). SEM images taken at 25kX, 50kX show that crystalline powder with the formation of agglomerates of size 300-400nm formed by nano powder of ZnO of 18-30mm..The crystallite size of the powder ranges between 18-27nm, when calculated using Scherer formula.**

***Keywords*— Combustion, Zinc nitrate, Ferric nitrate, Urea.**

I. INTRODUCTION

ZnO has a wide band gap of 3.37 eV and a large excitation binding energy of 60 meV, which can be obtained in a hexagonal wurtzite crystalline structure [1]. ZnO has various applications such as ultraviolet opto-electronic devices, transparent high power, high frequency electronic devices, piezo-electronic transducers and chemical gas sensors [2], conducting electrode in solar cells and thin-film transistor [3]. Doping with transitional metal elements leads to many interesting properties of ZnO. Currently, much experimental and theoretical research is based on dilute magnetic semiconductors (DMS) on ZnO doped with transition metal (TM) ions such as Mn, Fe and Co, since the predicted room temperature ferromagnetism in the DMS may be useful in spintronics[4].

Spintronics which is also known as spin electronics or Flextronics, it's the study of the intrinsic spin of the electron and its associated magnetic moment, in addition to its fundamental electronic charge, in solid-state devices.

Up to now, various approaches have been applied to prepare TM doped ZnO, such as sol-gel [5-9], thermal decomposition solid-state route [10-11], co-precipitation [12-13], chemical vapour deposition [14], solution combustion synthesis [15], by using different chemical precursors, Till now no one reported synthesis of ZnO doped with Fe using urea as fuel by solution combustion synthesis. In the present investigation, we have used the solution combustion technique to prepare transition metal Fe doped ZnO (Zn_{1-x} Fe_xO, where x = 0.01, 0.03, 0.05, 0.07, 0.1) powder using urea fuel because this technique is simple, fast, gives homogeneous product , involves low cost equipment and raw materials [16].

II. EXPERIMENTAL DETAILS

A. Raw materials used

Zinc nitrate hexa-hydrate ($Zn(NO_3)_2.6H_2O$) ferric nitrate nano-hydrate ($Fe(NO_3)_3.9H_2O$), Urea (NH_2CONH_2).

B. Solution Combustion Synthesis of ZnO

. The Fe doped ZnO (Zn_{1-x} Fe_xO , where x = 0.01, 0.03, 0.05, 0.07, 0.1 or 1%, 3%, 5% 7% 10% of Fe in Zn) powders were prepared by solution combustion process by using ferric nitrate and zinc nitrate as oxidizer and urea as fuel. The sample coding was done as per Table 1. The amount of chemicals were weighed as from the Table 2 and mixed in beaker. With adding distilled water the beaker was shaked untill it becomes transparent solution.The solution was taken in to the muffle furnace maintained at 500℃. Initially the chemicals boil for certain minutes to vaporise the water present in it. Then temperature of the solution increase simultaneously forming of froth. Then suddenly flame forms ending with the ash of desire composition with brown color.

The combustion reaction characteristics and the combustion details like expected product, yield obtained, and amount of chemicals taken and moles of chemical precursor and number of moles of fuel taken are shown in Table 2. The combustion details like nature of combustion combustion time, color of the flame, flame type, are shown in Table 1.

All obtained powders were characterized by XRD, Under the equilibrium conditions the reaction equations in these fuels nitrate systems for preparation of ZnO by urea fuel can be represented as below.

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$$(1\text{-}x)Zn\,(NO_3)_2.6H_2O + xFe\,(NO_3)_3 9H_20 + 1.66\,NH_2CONH_2 \rightarrow Zn_{(1-x)}\,Fe_x\,O + 1.66\,CO_2 + (2.66+0.5x)N_2 + (9.32+3x)H_2O \quad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

C. Phase analysis by X-ray diffraction (XRD**)**

X-ray diffraction studies were carried out for phase confirmation and calculating crystallite size of the milled samples, using D8-Advance-Bruker machine with Cu-K_α (wavelength of Cu-K_α (λ) ~1.5406 Å) radiations for all the measurements. Ni filter was used to attenuate K_β lines. The crystallite size of powders was calculated using Scherrer's formula.

$$d = K/\beta\cos\theta \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Where, β_θ is the full width at half maximum (FWHM) of diffracted peaks in degrees, d stands for the liner dimension of particles in meters, θ refers to Bragg's angle in degrees K' is the shape factor, generally known as a numerical constant and evaluated as 0.93 and depends on shape of crystallites.

III. RESULT AND DISCUSSION

Figure.1-5 show the XRD patterns of the synthesized 1, 3, 5, 7, 10% Fe doped ZnO ($Zn_{(1-x)}\,Fe_xO$ where X=0.01, 0.03, 0.05, 0.07, 0.1) powders respectively prepared by solution combustion synthesis. The patterns are found to be in good agreement with the standard peak positions of ZnO (JCPDS Card No. 36-1451). These peaks reveal that all the investigated samples are nano crystalline powder of hexagonal wurtzite structure. The crystallite size of Fe-doped ZnO powder was 18-27nm when calculated using Scherrer's formula and the data is given in Table 3.

Figure 6 shows SEM images of 0.01(1%) Fe doped ZnO powders. The powders were agglomerates in the size ranging between 300-400nm. The agglomerates were formed by crystallites of the size ranging between 18-30nm, which was in relevance to the crystallite size obtained using Scherrer's formula. Figure.7 shows the energy dispersive X-ray analysis (EDS) spectrum of ZnO confirming the presence of all elements of ZnO such as Zn, Fe, O.

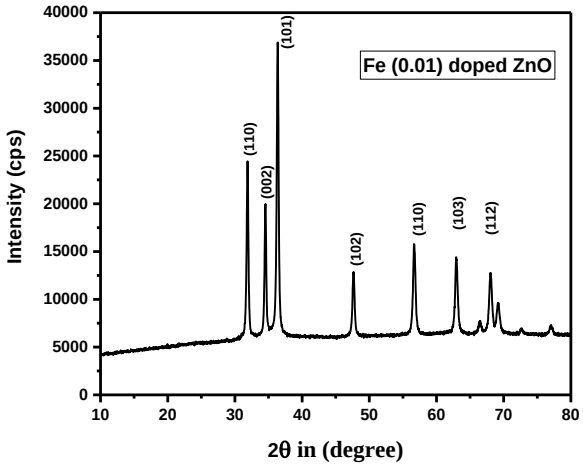

Fig. 1. XRD pattern of 1% Fe doped ZnO

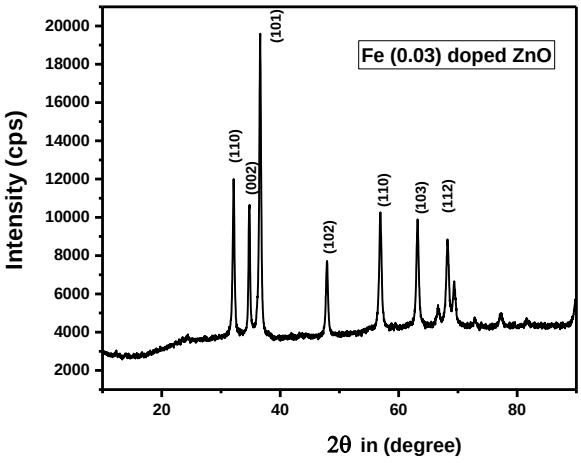

Fig. 2. XRD pattern of 3% Fe doped ZnO

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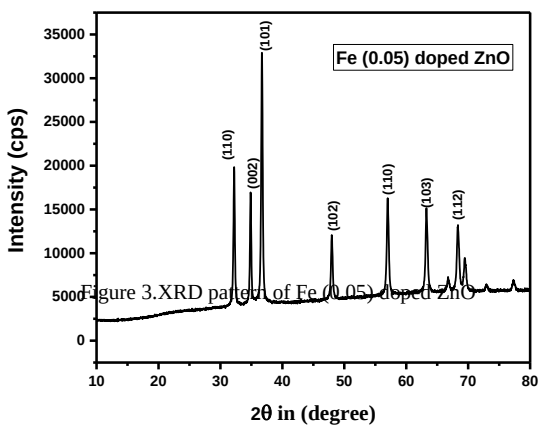

Figure 3.XRD pattern of Fe (0.05) doped ZnO

Fig 3.XRD pattern of 5% Fe doped ZnO

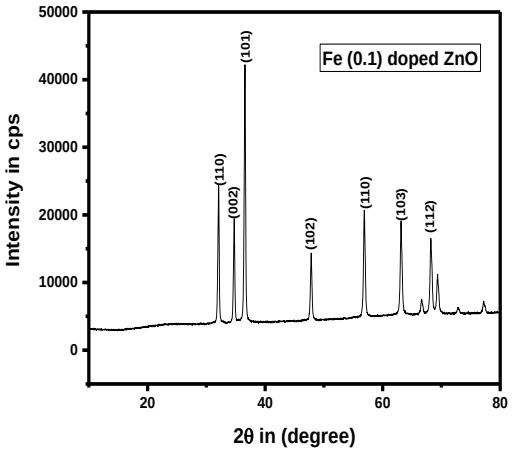

Fig 5. XRD pattern of 10% Fe doped ZnO

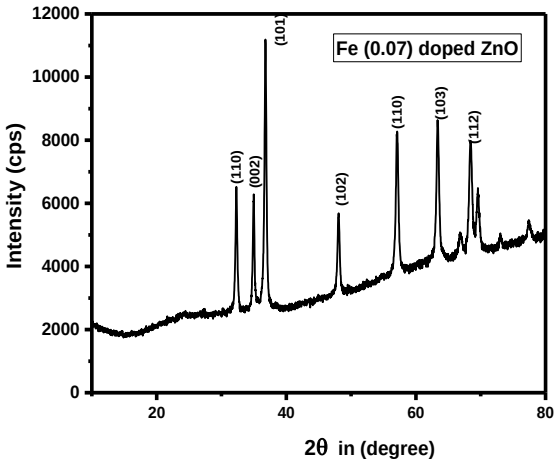

Fig 4. XRD pattern of 7% Fe) doped ZnO

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Table.1 Characteristics of the combustion reaction to produce ZnO

Sl No	Sample code	Doping %	Samples	Color	Combustion type	Combustion time
1	ZNH01	1	(2.94) $Zn(NO_3)_3.6H_2O$ + (0.029) $Fe(NO_3)_3.9H_2O$ + NH_2CONH_2	Brown	flame	5.42min
2	ZNH02	3	(2.88) $Zn(NO_3)_3.6H_2O$ + (0.08)$Fe(NO_3)_3.9H_2O$+NH_2CONH_2	Brown	flame	4.48min
3	ZNH03	5	(2.82) $Zn(NO_3)_3.6H_2O$+ (0.145) $Fe(NO_3)_3.9H_2O$+NH_2CONH_2	Brown	flame	5.23min
4	ZNH04	7	(2.76) $Zn(NO_3)_3.6H_2O$+ (0.20) $Fe(NO_3)_3.9H_2O$+NH_2CONH_2	Brown	flame	4.41min
5	ZNH05	10	(2.67) $Zn(NO_3)_3.6H_2O$+ (0.29) $Fe(NO_3)_3.9H_2O$+NH_2CONH_2	Brown	flame	5.09min

Table.2 Amount of urea fuel and oxidizers, used for the combustion reaction to produce ZnO

Sl No	Doping %	$Zn(NO_3)_3.6H_2O$ in (gm)	$Fe(NO_3)_3.9H_2O$ in (gm)	Fuel urea in (gm)	Expected powders in (gm)	Obtained powders in (gm)
01	1	2.94	0.04	1.0	0.8	0.89
02	3	2.88	0.12	1.0	0.8	0.78
03	5	2.82	0.2	1.0	0.8	0.69
04	7	2.76	0.28	1.0	0.8	0.63
05	10	2.67	0.4	1.0	0.8	0.62

Table 3.Effect of doping on Crystallite size

Doping %	2θ	β	θ	cosθ	$d = K\lambda / \beta \cos\theta$ in (nm)
1	36.37	0.42	18.185	0.9500	20.80
3	36.61	0.37	18.305	0.9494	23.63
5	36.50	0.32	18.25	0.9496	27.31
7	36.80	0.39	18.4	0.9488	22.43
10	36.58	0.47	18.29	0.9494	18.6

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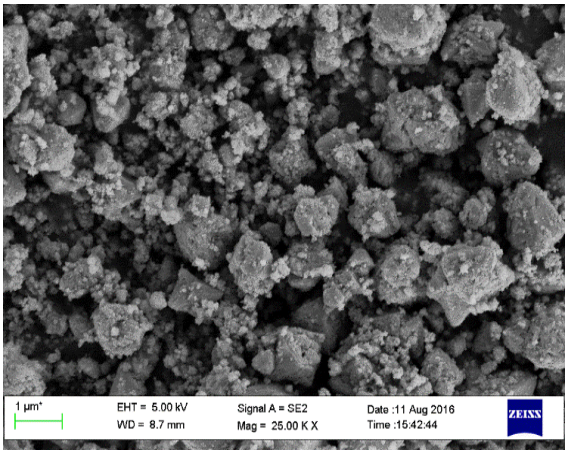
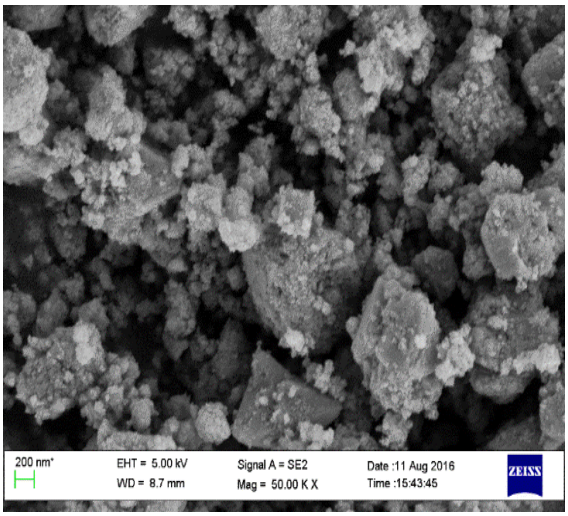


(a) Magnification=25 k



(b) Magnification=50 k

Fig 6. SEM images of 1% Fe doped ZnO (a) at 25 k Magnification (b) at 50k Magnification

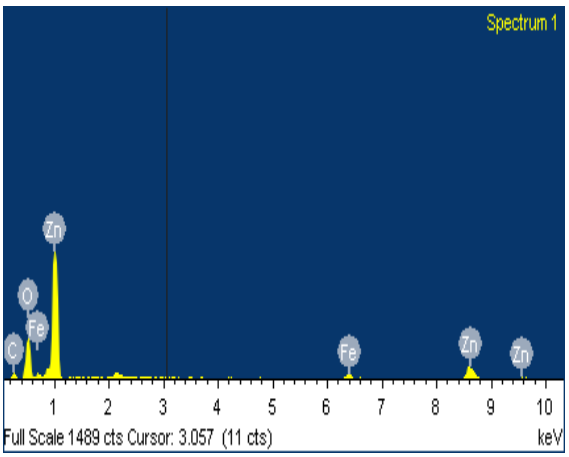


Fig.7 The EDS spectrum of (1%) Fe doped ZnO

CONCLUSION

For the first time Fe doped ZnO (Zn_{1-x} Fe_xO , where x = 0.01, 0.03, 0.05, 0.07, 0.1 or 1%, 3%, 5% 7% 10% of Fe in Zn) powders were synthesized by solution combustion method. The XRD patterns of all the samples show the formation of pure ZnO by comparing with standard JCPDS card no 36-1451. Up to 10% of Fe was soluble in ZnO. No impurity of iron oxide was present in XRD pattern. SEM images of the 1%Fe doped ZnO shows forrmation of aglomerate of ZnO of size 300 - 400 nm, formed by nano powder of size 18-30nm.

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