SYNTHESIS AND CHARACTERIZATION OF DOPED ZnO

Synopsis submitted in fulfilment of the requirements for the Degree of

DOCTOR OF PHILOSOPHY

By

GUNJAN SRINET

Department of Physics and Materials Science and Engineering
JAYPEE INSTITUTE OF INFORMATION TECHNOLOGY
(Declared Deemed to be University U/S 3 of UGC Act)
A-10, SECTOR-62, NOIDA, INDIA

August, 2014

SYNOPSIS

In order to achieve higher speeds, size of devices is diminishing continuously. As a result of this shrinkage, design parameters are impacted in such a way that materials in present use are pushed to their limits [1]. The alternative would be to exploit multifunctional properties of materials. Today, one of the hot topics among researchers is to use other unexploited degree of freedom, the spin of electrons, holes, nuclei or ions to gain new functionalities in both analog and digital electronics. The electronic charge, mass and spin form the foundation of present information technology. Integrated circuits and high frequency devices made of semiconductors, used for information processing, utilize the electronic charge. On the other hand the information storage is done by magnetic recording using electronic spin in a ferromagnetic metal [1]. But, in future, information technology may see semiconductivity (charge) and magnetism (spin) combined in a single device by exploiting both charge and spin to process and store the information. Such a device is called as "Spintronic device". It is difficult to realize such a device because semiconductors currently used in integrated circuits, transistors and lasers, such as silicon and gallium arsenide are nonmagnetic. Moreover, the magnetic fields that would be required to have a useful difference in the energy between the two possible electron spin orientations (up and down) are too high for everyday use [2].

So, the creation and control of spin-polarized currents in semiconductor is important. The integration of spintronics into existing semiconductor devices provided by ferromagnetic semiconductors is relatively easy. For instance, highly efficient spin injection is possible between semiconductor/semiconductor interface, whereas, due to conductivity mismatch, very less spin polarizations (a few percent) are possible between a ferromagnetic metal/semiconductor interface [3]. However, for realization of semiconductor-based spintronics, significant challenges related to the lifetime, control and detection of spin polarized carriers in semiconductors must be seriously addressed. Thus, semiconductors that can retain their ferromagnetism above room temperature are crucial to the practical application of spintronic devices.

One of the approaches to drive a semiconductor magnetic is introduction of magnetic ions such as Mn, Co, Cr and Fe into non-magnetic semiconductors. In these magnetic semiconductors, a small part of the lattice is made up of substitutional magnetic atoms (Figure 1). Hence, they are called diluted magnetic semiconductors (DMS) exhibiting both magnetic as well as semiconducting properties.

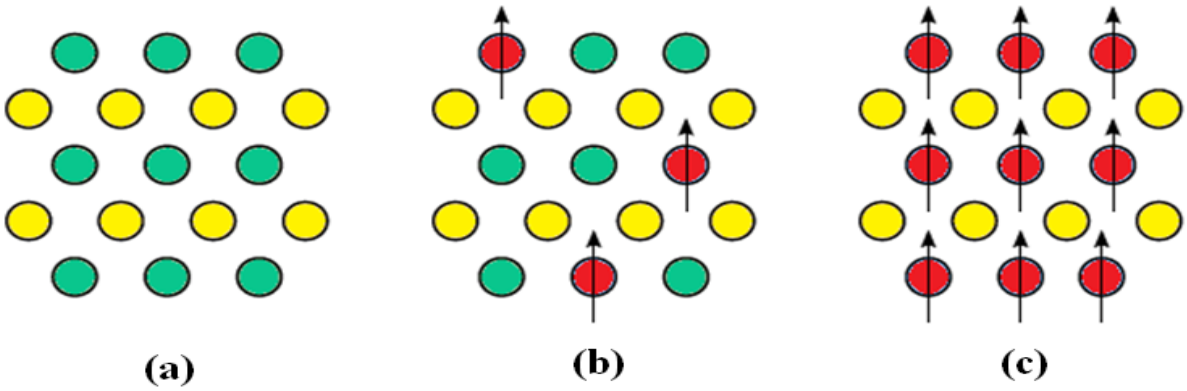

Figure 1: Illustration of different type of semiconductors: (a) nonmagnetic semiconductor, (b) Diluted magnetic semiconductor (c) Magnetic material.

DMSs typically have ordering temperatures lower than room temperature. There has been some success in achieving room temperature ferromagnetism (RTFM) in various semiconductors, but results are non-reproducible and controversial among various research groups. Investigation on DMSs was originally sparked by Ohno et al [4] for discovering Mn doped GaAs as low temperature ferromagnetic with Curie temperature (T_c) around 110 K. After theoretical predictions of RTFM in ZnO-based DMS systems by Dietl et al [5] and Sato et al [6], a number of groups investigated this phenomenon subsequently [7]. In spite of extensive studies, understanding the mechanism of magnetism of transition metal (TM) doped ZnO systems still remains a debatable issue, mostly due to the low reproducibility of results from samples prepared by different techniques [8, 9]. The RTFM observed for some samples has been attributed to different origins, including the original model of ferromagnetism mediated by free carriers or shallow donor electrons forming bound polarons [10] and alternative explanations such as the formation of secondary magnetic phases [11] and TM rich nanocrystals [12]. Under this scenario, careful experiments are needed to establish the intrinsic and robust magnetism.

Furthermore, ZnO is a highly promising and key technological material for a wide variety of next-generation applications due to its unique and versatile properties. As a wide gap (3.37 eV at 300 K) semiconductor, ZnO is an ideal candidate for optoelectronic devices laser diodes (LDs) and white light emitters with important applications in high density data storage systems and solid-state lighting [13–16]. Few optical properties of ZnO overlap with GaN which is widely used in production of ultraviolet, blue, green and white light emitting devices. But, it has some advantages over GaN which include a large exciton binding energy

(~ 60 meV), good radiation hardness, availability of high quality ZnO bulk single crystals and relatively low cost for ZnO based devices. It also exhibits good luminescence properties and one can tune the band gap of ZnO which makes it more applicable for devices.

There are several reports available in the literature on optical and magnetic properties of transition and non-transition metal doped ZnO [17, 18], but still it is a good candidate for improving ferroelectric (FE) properties, which are of considerable importance in low dimensional ferroelectrics because of the demand for non-volatile FE memory device miniaturization [19, 20]. Generally, pervoskites are used for FE memory devices which are structurally complicated and difficult to synthesize [21, 22]. There are only few ZnO based FE materials reported in the literature which are highly controversial. Yadav et al. [23] observed dielectric anomaly at 430-460 K and claimed the anomaly not to be ferroelectric, Gupta et al. [24] reported ferroelectric transition around 343 K in K doped ZnO nanorods, Yang et al. [25] observed and explained the multiferrioc behaviour of Cr doped ZnO. Recently, Gupta et al. [26] observed low ferroelectric phase transition at 69°C.

Under this scenario, the general aim of this thesis is to extend the understanding of few key issues in the current research progress of ZnO. These are in particular, to achieve RTFM in TM doped ZnO systems and to search the possible mechanism behind it. For the realization of ZnO based optoelectronic devices and the improvement of material quality, we focused on band gap tuning and enhanced photoluminescence properties with doping of metal ions. Along with this, we also studied the dielectric properties of doped ZnO systems. Brief sketch of the proposed thesis is as follows:

Chapter I gives general introduction of spintronics, diluted magnetic semiconductor and ZnO. As magnetic properties of ZnO form the subject matter of the thesis, so, properties of diamagnetic, paramagnetic, ferromagnetic and anti-ferromagnetic materials are introduced in brief. Fundamental interactions which are believed to govern the magnetic properties like RKKY interaction, the mean field zener model, bound magnetic polaron (BMP) theory, etc. are discussed in detail. Next section of this chapter is devoted to recent progress in optical and magnetic properties of ZnO and a review of critical issues for realization of ZnO-based spintronic and optoelectronic devices. In the last section, some potential semiconductor-based spintronic devices are briefly summarized.

Chapter II describes synthesis routes used during the present work which includes the standard solid state route for Mn and Ni doped ZnO systems, sol-gel route for Mn, Ni and Co

doped ZnO systems and thermal decomposition method for preparation of nanoparticles of Cr, Al, Mg and Ba doped ZnO. The chapter also covers a comprehensive discussion of different experimental techniques used for the characterization in the present work. The characterization techniques include X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infra-red (FTIR) spectroscopy, superconducting quantum interference device (SQUID), UV–Visible spectrometer, luminescence spectrophotometer etc.

Chapter III deals with a comparative study of two systems of Mn doped ZnO systems which were prepared by solid state (series S1) and sol-gel (series S2) route. The refinement of the unit cell parameters revealed that cell parameters slightly increased with Mn concentration, indicating the substitution of divalent Mn^{2+} (0.66 Å) ions on Zn^{2+} (0.60 Å) ions in ZnO crystal lattice for both S1 and S2 series. The band gap increased monotonically with Mn concentration for both series which can be explained by the Burstein-Moss effect. PL spectra show that the vacancy states have increased for series S2 which can strongly affect magnetic properties. For series S1, weak hysteresis loops were observed for samples with the lower Mn concentration which manifest the slight ferromagnetic ordering at room temperature. However, ferromagnetism completely disappeared for the sample with 6% Mn concentration. Magnetic measurements clearly indicate that samples prepared by sol-gel route (S2) show better magnetic properties in comparison to samples prepared by the solid state route. The MH curves of the series S2 were tried to fit on the BMP model, but the calculated concentration of BMP ($\sim 10^{14}$ cm^{-3}) appears to be quite low to achieve percolation. For further investigations of magnetic ordering in S2 samples, we tried to explore it with the help of modified Curie Weiss law and observed some antiferromagnetic ordering in the samples.

(Results of this work are published in (i) Journal of Materials Science: Materials in Electronics, vol. 25, pp. 3052, 2014).

Chapter IV presents the synthesis and characterization of Co doped ZnO samples prepared by sol-gel route (series S3). For series S3, the X-ray diffraction, X-ray photoelectron spectroscopy and UV–visible spectroscopy confirmed the substitution of Co ions on Zn sites without changing the wurtzite structure. No segregated secondary phases or Co rich clusters were detected. Optical absorption spectra of the samples exhibit a blue shift in the absorption band edge with increasing dopant concentration. Photoluminescence measurements show a

blue shift in UV emission peak with the increase in Co concentration and a slight shift in the green emission band at around 509 nm which gets suppressed for higher sintering temperature. The field dependence of magnetization observed at room temperature exhibits clear ferromagnetic behavior. Efforts have been made to fit the experimental M–H data using the magnetic polarons model (BMP) which involves localized carriers and magnetic cations. The calculated concentration of the BMPs is found to be below the typical percolation threshold in ZnO. Thus BMP model alone is not sufficient to explain the RTFM behaviour in ZnO. To know the exact magnetic ordering in the system, we also attempted to fit temperature dependent magnetization curves with the Curie–Weiss Law which shows antiferromagnetic ordering in all samples.

(Results of this work are published in (i) Ceramic International, vol. 39, pp. 6077, 2013, (ii) Material Science in Semiconductor processing, vol. 15, pp. 314, 2012).

In **Chapter V,** Ni and Cr doped ZnO systems were studied in detail. X-ray diffraction pattern of Ni doped ZnO (series S4) suggested an appearance of a secondary phase of NiO only in 6% Ni doped sample. Phonon modes in Ni doped ZnO nanoparticles were studied through FTIR measurements. Furthermore, the enhancement in optical band gap with Ni doping from 3.29 to 3.32 eV has been observed through UV-visible spectroscopic analysis. Photoluminescence spectra of $Z_{1-x}Ni_xO$ show the UV-emission peak showing the blue shift with increase in doping concentration followed by broad visible (blue) emission corresponding to the defect emission whose intensity decreased with increasing Ni concentration. A clear RTFM is observed in all samples but saturation magnetization decreased with increasing Ni content. The suitability of bound magnetic polarons (BMP) model is checked and numbers of BMPs are found to be of the order 10^{15} per cm^3, which is very small for the percolation in ZnO. In the present case, oxygen rich stoichiometry with enhanced Zn-O bonding favours the indirect Ni-O-Ni ferromagnetic exchange coupling and reduction of oxygen vacancies leading to strong hybridization of Ni in ZnO host matrix responsible for RTFM.

Interestingly, band gap decreased with Cr doping (series S5) from 3.23 to 3.04 eV, this band gap narrowing has been interpreted in terms of the s, p–d spin-exchange interactions between delocalized s- or p-type band electrons of Zn and O atoms, respectively and localized d electrons of transition metal replacing the cation. This shift is most probably occurs due to

band structure deformation by Cr ions doping in the lattice of ZnO structure. The estimated number of BMPs is higher than earlier observed values in other TM doped systems and it is above the threshold of percolation in DMS and this is quite likely due to the high value of magnetization of the present system.

(Results of this work are published in (i) Journal of Applied Physics, vol. 114, pp. 03391 2013, (ii) AIP conference proceeding, vol. 1512, pp. 1282, 2013, (iii) AIP Conference Proceedings, vol. 1591, pp. 1476, 2014).

In **Chapter VI,** our main focus was on the structural and optical properties of alkaline metal (Al, Mg) doped ZnO nanoparticles prepared by thermal decomposition method (series S6 and S7). X-ray diffraction studies confirmed the substitution of Al and Mg on Zn sites without changing the hexagonal structure of ZnO. Also, lattice parameters, the crystallite size and other physical parameters such as strain, stress and energy density were calculated from various modified form of W-H equations and their variation with the doping of Al is discussed. A blue shift in the energy band gap attributed to increase in carrier concentration (Burstein Moss Effect) is observed by absorption spectra with Al and Mg. Photoluminescence studies show a strong and dominant peak corresponding to the near band edge emission in ultra violet range and a broad band in the range 420-700 nm corresponding to defects and oxygen vacancies. From the FTIR spectra, we observed that the effective mass of Zn (Al)-O bond decreased with Al substitution because of lower atomic weight of Al than Zn. Also, the average force constant decreased with Al substitution which results an increment in the average Zn (Al)–O bond length. Besides that, opposite behaviour was obtained in the FTIR measurement of Mg doped ZnO nanoparticles. The tunability of the band gap of ZnO nanoparticles could eventually be useful for potential optoelectronic applications.

(Results of this work are published in (i) Ceramic International, vol. 40, pp. 4025, 2014. (ii) AIP Conference Proceedings, vol. 1536, pp. 247, 2013).

Chapter VII is devoted to the dielectric and ferroelectric properties of Ni and Ba doped ZnO samples (series S8 and S9) prepared by different routes.

X-ray diffraction confirmed the appearance of NiO phase for 6% Ni doping. Significant blueshift with Ni doping was observed in UV–visible studies, strongly supported by

photoluminescence spectra that show a high intensity UV emission peak followed by the low intensity green emission band corresponding to oxygen vacancies and defects. The photoluminescence analysis suggested that doping of Ni can affect defects and oxygen vacancies in ZnO and give the possibility of band gap tuning for applications in optoelectronic devices. High values of dielectric constant at low frequency and a strong dielectric anomaly around 320°C were observed.

For series S9, the substitution of Ba on Zn sites of the wurtzite structure of ZnO was also observed by XRD pattern. FESEM images show some structural transformation in the morphology of nanostructure with Ba doping. The IR bands corresponding to Zn show a variation in the vibrational frequencies after Ba doping which may be due to the difference in ionic radii of Zn and Ba as well the structural changes induced due to doping. Band gaps are observed to be 3.18 eV for pure and 3.14 eV for 5% Ba doped ZnO, respectively. Red shift in band gap is observed in the UV-Visible spectra after Ba doping, supported by photoluminescence spectra and showing enhanced defect states with Ba doping. In dielectric studies, high value of dielectric constant and transition temperature at (~ 330°C) were observed. High value of remnant polarization (1.01 μCcm^{-2}) and low value of coercive field (2.02 $kVcm^{-1}$) were also observed in ferroelectric studies which can be useful for potential applications.

(Results of this work are published in (i) Ceramic international, vol. 39, pp. 7557, 2013, (ii) Materials Letters, vol. 126, pp. 274, 2014).

In **Chapter VIII,** a summary of research work is given.

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[1] **Srinet G.**, Kumar R. and Sajal V., "*High T_c ferroelectricity in Ba-doped ZnO nanoparticles"*, Materials Letters, vol. 126, pp. 274-277, 2014. (**Thomson Reuters I. F.** = 2.26, **h-index** = 83, **h5-index =** 46, **Published by** Elsevier, **Indexed in** SCI and SCOPUS).

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Workshops/schools Attended

[1] Participated in "IUAC School of thin films", at Inter-University Accelerator Centre, New Delhi from, 11-13 December-2012.

Gunjan Srinet
(Research Scholar)

Dr. Ravindra Kumar
(Supervisor)