

**SPRAYED LOW COST, HIGHLY CONDUCTING AND TRANSPARENT BORON (B)  
AND NITROGEN (N) CO-DOPED ZnO THIN FILMS: SYNTHESIS AND PHYSICAL  
PROPERTIES**

*A final synopsis in candidature for the degree of Doctor of Philosophy*

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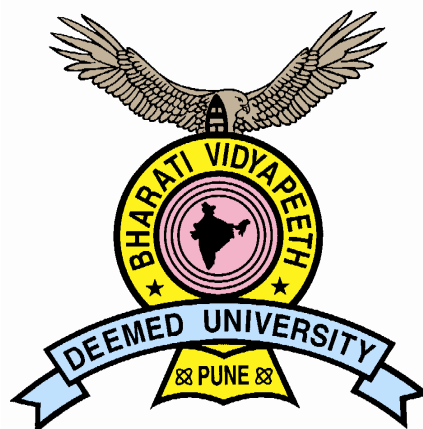
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## 1. INTRODUCTION

In the past few years, transparent conductive oxide (TCO) thin film has been widely used as a transparent electrode in several optoelectronic devices [1]. TCO films are generally deposited on glass substrates used as window electrode for solar cells or liquid crystal displays. Certain applications of the opto-electric devices call for the growth of TCO films on flexible or plastic substrates [2, 3]. The most commonly used TCO thin films include Sn-doped  $\text{In}_2\text{O}_3$  (ITO) thin films, F-doped  $\text{SnO}_2$  (FTO) thin films, and Al-doped ZnO (AZO) thin films and recently studied BN:ZnO thin films. Among these TCO thin films, codoped ZnO thin films have attracted considerable attention as substitutes for other TCO materials because of their high transmittance in the visible region, low resistivity, low cost and abundancy, nontoxic nature, easy fabrication, and high stability in hydrogen plasma [3–6].

Today ZnO is one of the hottest research fields in advanced materials and devices, emerging as one of the most important electronic and photonic materials with great potential applications in information technology, biotechnology, nanoscale science and engineering [7-9]. ZnO has been extensively studied for its promising applications in opto-electronic devices such as light emitting diodes (LEDs) and laser diodes (LDs), because of its wideband gap of 3.37 eV and large exciton binding energy of 60 meV at room temperature [10,11]. For many advanced applications, the development of ZnO based devices such as  $p$ - $n$  homo-junctions can be realized by utilizing both  $n$ -type and  $p$ -type codoped ZnO films. Generally, trivalent cation dopants including Al, B, Ga and Mo were explored to release free electrons and therefore provided a wider band gap [12-14]. From those dopants, the ionic size of  $\text{B}^{3+}$  is more closing to  $\text{Zn}^{2+}$  and the bond length of B-O is much smaller than that of Zn-O, indicating better opto-electric properties.

The  $p$ -type doping in ZnO can be realized by substituting either group V elements (N, P, As, and Sb) for O sites or group I elements (H, Li, Na, Ag and K) for Zn sites [15-19]. Among these acceptors, nitrogen (0.146 nm) is the most suitable dopant because of its nearly equal ionic radius to oxygen (0.132 nm). Although considerable efforts have been taken to realize N-doped  $p$ -type ZnO and ZnO based  $p$ - $n$  diodes. But it is still difficult to achieve reproducible and good quality  $p$ -type conduction in N-doped ZnO [20, 21]. There have been several reports on the growth of dual acceptor  $p$ -type ZnO films such as dual acceptor doping of Li-N [22], Ag-N [23]

and K–N [24] by various techniques. In recent years, Ti, Ag, or In have been used as dopants to enhance the opto-electric performances of ZnO based films [25–29].

However, achieving *p*-type ZnO impedes the development of advanced opto-electrical devices. ZnO occurs naturally as an *n*-type semiconductor due to a large number of intrinsic defects such as oxygen vacancies ( $V_O$ ), Zn interstitials ( $Zn_i$ ) and Zn anti-site defects ( $Zn_O$ ). Therefore, it is very difficult to form the shallow acceptor levels because these acceptors can be compensated by numerous ZnO native defects, resulting in the formation of deep donor level traps [30]. These are attributed to the self- compensation of excess N atoms in the ZnO films due to the relatively low solubility limit and structural mismatches such as a lattice mismatch of 18% and a thermal mismatch of 29% [31, 32]. As a result, these structural defects passivate incorporated N atoms and kill ionized free electrons, which hamper the *p*-type conductivity in the ZnO films [33, 34].

To improve the properties of transparent conductors, codoping strategies are used. For example, Jiang *et al.* [35] prepared Al-Ti codoped ZnO films by RF sputtering and obtained highly conductive films with a minimum resistivity of  $7.96 \times 10^{-4} \Omega\text{-cm}$ . The most notable result is obtained for Al–Co codoped ZnO films prepared by a sol–gel method, in which resistivity as low as  $3.5 \times 10^{-4} \Omega\text{-cm}$  and high mobility as high as  $50 \text{ cm}^2/\text{Vs}$  are reported [36]. Whereas nitrogen is regarded as the best candidate to induce *p*-type doping in boron doped ZnO thin films by co-doping process, because with the first-principle calculations Cui *et al.* have investigated the *p*-type nature of boron and nitrogen codoped ZnO [37]. A *p*-type B-N codoped ZnO films were grown on quartz by magnetron sputtering and post-annealing techniques with room-temperature resistivity of  $2.3 \Omega\text{-cm}$ , Hall mobility of  $11 \text{ cm}^2/\text{Vs}$  and carrier concentration of  $1.2 \times 10^{17} \text{ cm}^{-3}$ . Using this they have fabricated the ZnO homojunction by deposition of an undoped *n*-type ZnO layer on the B–N codoped *p*-type ZnO layer showed clear *p–n* diode characteristics [38,39].

## 2. SCIENTIFIC OBJECTIVES OF THE THESIS

(i). To use cost effective, simple chemical method : Spray pyrolysis method to synthesize ZnO thin films and optimization of various process parameters like air-flow rate, volume of precursor, precursor concentration and substrate temperature to obtain well adherent, uniform, transparent and conducting films on simple microscopic glass substrate.

- (ii). To use above process parameters to optimize and obtain boron-doped highly transparent and conducting ZnO thin films (B:ZnO). To study the effect of variation in dopant concentration and substrate temperature variation on physical and chemical properties of B: ZnO thin films
- (iii). To obtain nitrogen-doped highly transparent and conducting ZnO thin films (N:ZnO) by spray pyrolysis technique. To investigate the role of variation in dopant concentration and substrate temperature on physico-chemical properties of nitrogen doped ZnO thin films.
- (iv). As discussed previously N dopant in ZnO is highly unstable due to self- compensation of acceptors it was aimed to obtain highly transparent and conducting N:ZnO thin films with optimization of film properties. Also the morphological and electrical evidence is investigated to study the ageing effect of N:ZnO thin films.
- (v). To synthesize boron and nitrogen codoped ZnO thin films (BN:ZnO) with variation in boron dopant concentration, nitrogen dopant concentration and substrate temperature to obtain highly transparent and conducting properties. Well focus is aimed to obtain *p*-type conducting BN: ZnO thin films with reduction of all acceptors and donors compensation effect.
- (vi). Study of physico-chemical properties of boron nitrogen codoped ZnO thin films (BN:ZnO) to analyze the transparent and conducting nature of the films.

### 3. THESIS OUTLINE

The present thesis comprises of seven chapters and is assembled in following orders;

In **Chapter I**, we have planned to provide brief summary of TCO materials with particular focus on as deposited ZnO and doped ZnO TCO materials. Also it focuses on the study of basic physical properties along with applications of ZnO materials with particular defect study and n-type and p-type ZnO properties review.

**Chapter II**, will comprises two sections, first section covers the study of spray pyrolysis deposition process with their mechanism, advantages and apparatus details and the second section covers the detail about various physico-chemical characterization techniques employed in present study which includes X-ray analysis, morphological elucidation with scanning electron Microscopy (SEM), atomic force microscopy (AFM) and transmittance electron microscopy images (TEM). These films also characterized for their optical analysis using Uv-vis Spectroscopy, electrical analysis using Hall measurement, elemental analysis using EDAX analysis, XPS analysis and the depth profile of film by using SIMS spectroscopy.

In **Chapter III** the basic optimization of all the process parameters related to the spray pyrolysis with the aim of obtain highly transparent and conducting ZnO thin film. With the same aim main process parameters such as airflow rate is varied from 3lpm to 9lpm, volume of precursor is varied from 20ml to 70ml, concentration of precursor varied from 0.3M to 0.7M and substrate temperature is varied from 300°C to 500°C. The variation in all process parameters gives formation of highly transparent and conducting film formation at 7lpm air-flow rate, 50ml volume of precursor with 0.5M precursor concentration and 450°C substrate temperature.

It revealed hexagonal wurtzite crystal structure having orientations along (100), (002), (101) and (110) planes and shows crystallite size to be 42.17nm. The ZnO grain growth over glass substrate is uniform and spherical with optical transmittance ~80%, band gap of 3.23eV, having *n*-type electrical conductivity with electrical resistivity  $1.763 \times 10^{-2} \Omega\text{-cm}$ , hall mobility  $39.89 \text{ cm}^2/\text{Vs}$  and carrier concentration of  $13.37 \times 10^{19} \text{ cm}^{-3}$ . From the TEM several nanometer-sized spherical and non-spherical crystals were observed in agglomerate form having average grain size of ~25nm. The SAED pattern of optimized ZnO thin film shows polycrystalline nature of films. The estimated amounts of atomic weight percentage of Zn and O in deposited thin films by EDX spectra are approximately 71.84% and 28.15%. The clear indication of Zn and O binding energy peak at 1021.12eV for Zn 2p<sub>3/2</sub> and at 529.21eV for O1s spectrum shows formation of ZnO thin film.

The **Chapter IV** discuss the formation of boron doped ZnO (B:ZnO) thin films to enhance the *n*-type conductivity of ZnO thin films. The 2 at% boron doped ZnO thin film deposited at 450°C substrate temperature by spray pyrolysis technique reveals highly transparent and *n*-type conducting film formation. The XRD spectra of such film reveals diffraction oriented along (002) peak located at the angular position  $2\theta = 34.53^\circ$  giving crystallite size of 29nm. This peak is characteristic of the hexagonal close packed structure of this material, in agreement with the standard position ( $2\theta = 34.5^\circ$ ). The SEM image depicts well-covered, uniform deposition over the film surface consisting of nearly spherical grains with agglomerated base surface of approximately 30 to 40 nm size. The AFM images revealed that boron doped ZnO thin films possess surface roughness of 338.427 nm. The boron doped ZnO thin film employed highly transparent in the visible range (400–750 nm) of the optical spectrum, giving maximum of 95% at 550 nm with band-gap energy of 3.325 eV. The lowest resistivity  $1.846 \times 10^{-3} \Omega\text{-cm}$  with carrier concentration of  $8.52 \times 10^{21} \text{ cm}^{-3}$  was obtained. The Hall-mobility of  $45.97 \text{ cm}^2/\text{Vs}$  with

negative hall coefficient ( $-6.28 \text{ m}^3/\text{C}$ ) of boron doped ZnO thin film showed good *n*-type conductivity of films. The high resolution of XPS spectra and the depth profile using SIMS spectroscopy represents a strong proof presenting detection and effect of incorporation of  $\text{B}^{+3}$  at  $\text{Zn}^{+2}$  sites.

In **Chapter V** we have discussed about the N;ZnO thin film revealing *p*-type conductivity with 0.4M nitrogen concentration and 450°C substrate temperature formed and studied with physico-chemical characterization and ageing effect. With XRD it is confirmed that film reveals reflection peaks (100), (002), (101), (110), (200), (112) and (201) of the N:ZnO can be indexed as pure hexagonal ZnO with cell parameters  $a = 3.249 \text{ \AA}$  and  $c = 5.206 \text{ \AA}$ . The shells growth with sharp pointed edges and flattened at the midpoint indicates that nitrogen is incorporated in the ZnO film. The optical transmittance of N:ZnO thin film is of order of  $\sim 90\%$  (550nm). The indirect band gap found to be 3.232eV was due to the decrease of gap between conduction band edge and valance band edge (semi conducting property) of the zinc oxide film by the nitrogen incorporation. With the Hall probe measurement N:ZnO thin film shows best compromise between the lowest resistivity, the highest mobility and carrier concentration having resistivity  $\rho = 4.126 \times 10^{-3} \text{ } \Omega\text{-cm}$ , a mobility  $\mu = 12.39 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  and a carrier concentration  $n = 4.47 \times 10^{18} \text{ cm}^{-3}$  (Hall coefficient  $R_H = +2.65 \text{ m}^3/\text{C}$ ). The TEM digital photoimage shows agglomerated-structure, superposition of spheres and shells, are identified which consist of small equiaxed nano-particles that are heavily agglomerated into large secondary particles. The XPS spectrums; the N 1s region near 400 eV, the Zn  $2p_{3/2}$  region near 1020 eV and the O 1s region near 529 eV. The appearance of the 396.1 and 403.9 eV peaks has confirmed that both  $(\text{N})_O$  and  $(\text{N}_2)_O$  are present in ZnO:N films.

Also in the chapter for quality and durability point of view further the morphology and conductivity of N:ZnO thin films is measured and results indicates the morphological and electrical proof for escape of nitrogen through the ZnO films due to its highly unstability. We observed that, after 30 days (one month) the SEM micrograph reveals same crystal morphology in the form of shells but it was observed that some shells were opened at the edge. The sample shows increase in resistivity of N:ZnO thin film from  $4.128 \times 10^{-3} \text{ } \Omega\text{-cm}$  to  $0.3247 \text{ } \Omega\text{-cm}$  and carrier concentration is decreased to  $1.26 \times 10^{17} \text{ cm}^{-3}$  from  $4.47 \times 10^{18} \text{ cm}^{-3}$ . Also the *p*-type conductivity is changed to *n*-type due to reduction in acceptors in the film.

In **Chapter VI** we have focused our intention mainly to form highly transparent and conducting boron nitrogen codoped ZnO thin films by spray pyrolysis technique. The boron nitrogen codoped ZnO thin film formed with 2 at% boron and 0.4M nitrogen at 400°C by simple spray pyrolysis deposition technique reveals transparent and *p*-type conducting nature. The XRD pattern of BN:ZnO indicates three reflection peaks of hexagonal wurtzite structure (002),(101), and (201) at  $2\theta = 34.58, 36.41$  and  $31.9$  respectively represents a preferred orientation along the *c*-axis perpendicular to the substrates. The surface morphology of BN:ZnO films deposited at 400°C using spray pyrolysis underwent inhomogeneous growth and became closely compact with a rough surface reveals grain growth like human vertebrae column with large and visible grain boundaries. It can be noticed that the BN:ZnO thin film has visible transmittance (400–800 nm) over 90% due to formation interference over the substrate surface. The estimated value of band gap for BN:ZnO thin film is  $\sim 3.26$  eV. The lowest resistivity of  $2.39 \times 10^{-2} \Omega\text{-cm}$ , highest carrier concentration  $5.62 \times 10^{18} \text{cm}^{-3}$  with hall mobility of  $0.34 \text{cm}^2/\text{Vs}$  shown by the BN:ZnO thin film forming *p*-type conductivity. The chemical bonding states of N and B in the films were examined by XPS analysis for the 0.4M N:Zn<sub>0.98</sub>O<sub>B<sub>0.02</sub></sub> sample showing clear peaks for N<sub>O</sub> and (N<sub>2</sub>)<sub>O</sub> at 395.28 eV and 404.75 eV respectively. Therefore, we assigned N 1s peak around 399.23 eV for NO–Zn complex. The SIMS depth profile of B, N, Zn and O in the B–N codoped ZnO film deposited in 2 at% boron concentration and 0.4M nitrogen concentration gives evidence that B and N have been clearly detected, and their concentration profiles are quite flat throughout the film depth scanned to depth of 100nm.

In **Chapter VII** we conclude our study on the basis of derived results for the approach of enhancing transparent and conducting boron nitrogen codoped ZnO thin films formation for application of TCO films.

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### List of publication in refereed journals

- 1) Rajendra S. Gaikwad, Gauri R. Patil, Bhagwat N. Pawar, Rajaram S. Mane, Sung-Hwan Han, Liquefied Petroleum Gas Sensing Properties of Sprayed Nanocrystalline Zinc Oxide Thin Films, **Sensors and Actuators : A Physical**, 189 (2013) 339-343
- 2) Rajendra S. Gaikwad, Sambhaji S. Bhande, Rajaram S. Mane, Bhagwat N. Pawar, Sanjay L. Gaikwad, Sung-Hwan Han, Oh-Shim Joo, Roughness-based monitoring of transparency and conductivity in boron-doped ZnO thin films prepared by spray pyrolysis, **Material Research Bulletin**, 47 (2012) 4257-4262
- 3) Rajendra S. Gaikwad, Rajaram S. Mane, Bhagwat N. Pawar, Rohan B. Ambade, Hee Joon Ahn, Sung-Hwan Han, Oh-Shim Joo, Nitrogen-doped ZnO shells: Studies on optical transparency and electrical Conductivity, **Materials Research Bulletin** 47 (2012) 1246–1250
- 4) Rajendra S. Gaikwad, Gauri. R. Patil, Mahesh. B. Shelar, Bhagwat. N. Pawar, Rajaram. S. Mane, S. H. Han, and Oh. Shim. Joo, Nanocrystalline ZnO Films Deposited by Spray Pyrolysis: Effect of Gas Flow Rate **International Journal of Self Propagating High Temperature Synthesis**, 21 (2012) 178–182.
- 5) Gauri. R. Patil, Rajendra S. Gaikwad, Mahesh. B. Shelar, Rajaram. S. Mane, S. H. Han and Bhagwat. N. Pawar, Role of concentration and temperature on well aligned ZnO nanorods by low temperature wet chemical bath deposition method, **Scholars Research Library, Archives of Physical Research**, 3 (5) (2012) 401-406
- 6) Rajendra S. Gaikwad, Rajaram. S. Mane, Bhagwat. N. Pawar, S. Ravichandran, S. H. Han, Oh. Shim Joo & V. Sudhakar, Studies on Nanostructured ZnO thin films deposited by spray pyrolysis, **Asian Journal of Biochemical and Pharmaceutical Research**, 1 (2011) 391-398
- 7) S. S. Shinde, Prakash S. Patil, Rajendra S. Gaikwad, Rajaram. S. Mane, Bhagwat. N. Pawar, K. Y. Rajpure, Influences in high quality zinc oxide films and their photo-electrochemical Performance, **Journal of Alloys and Compounds**, 256 (2010) 1-8
- 8) Bhagwat. N. Pawar, Rajendra S. Gaikwad, Gauri R. Patil and Rajaram. S. Mane, Molarities Dependence Deposition and Characterization of Chemical Sprayed Metal Oxide (ZnO) Thin Films, **Bharati Research Journal** 7 (2012) 1-6

### **Publications in Process:**

1. Sustainable transparent and conducting Boron Nitrogen codoped ZnO thin films by spray pyrolysis technique, **Solar Energy Materials and Solar cells** (submitted)
2. Synthesis of nanostructural, optical and liquefied petroleum gas sensing of Boron doped SnO<sub>2</sub> films by spray pyrolysis, **Sensors and Actuators: B chemical** (submitted)
3. Development of transparent and metallic ZnO nanocrystals for sensing carbon dioxide and carbon monoxide gas, **Journal of Applied Surface Science** (submitted)
4. Transparent and conducting *p*-type ZnO: Nitrogen temperature dependent optoelectronic properties. **Journal of Physics D: Applied Physics** submitted.
5. Studies of Structural, Optical and Magnetic Characterization of Zinc Oxide Thin Films **Journal Of Applied Surface Science** submitted

### **Conference / Seminar / Meeting / Workshop / School / Training in International and National levels.**

1. International workshop on Nanotechnology and Advanced Functional Materials (NTAFM-2013) at National Chemical Laboratory, Pune, during 24-25<sup>th</sup> July, 2013.
2. International Conference on Solar Energy Photovoltaic's (ICSEP-2012) at the School of Electrical Engineering, KIIT University Bhubaneswar, Orissa on 19<sup>th</sup> to 21<sup>st</sup> Dec, 2012
3. Indo-German Workshop on Advanced Materials for Future Energy Recruitments (WAMFER-2012), organized by Department of Physics & Astrophysics, University of Delhi and Max-Planck Institute of Polymer Research, Mainz, Germany on 29<sup>th</sup> Nov-1<sup>st</sup> Dec, 2012
4. International Conference on Advances in Polymeric Materials and Nanotechnology (POLYTEC-2012) at BVDU, Poona College of Pharmacy, Pune during 15<sup>th</sup> -17<sup>th</sup> Dec, 2012
5. International Conference on Nanoscience and Nanotechnology, at Swami Ramanand Tirth Marathwada University, Nanded on 11-14<sup>th</sup> Jan 2011
6. International conference on nanoscience and nanotechnology (ICNN-2011), Coimbatore Institute of Technology - India and Cinvestav– Mexico during 6<sup>th</sup> – 8<sup>th</sup> July 2011
7. International Conference on: Nanotechnology Materials and Composites for Frontier Applications at Bharati Vidyapeeth Deemed University, Pune during 14<sup>th</sup>-15<sup>th</sup> Oct, 2010

8. International Workshop on Nanotechnology and Advanced Functional Materials, at National Chemical Laboratory, Pune during 9<sup>th</sup> -11<sup>th</sup> July 2009
9. Transparent and Conducting Sprayed Nanocrystalline ZnO Thin Films, Raman conference for Research Students, SRTMU, Nanded on 5<sup>th</sup> March, 2010
10. National conference on Advances In Science and Technology of Nonmaterial's, Sakari, Dhule. 8<sup>th</sup> -9<sup>th</sup> March, 2010
11. Two days National Workshop on Recent Advances in Material Engineering and Technology (MET-2012) at SKN, Sinhgad Institute of Technology and Science, Lonavala during 2<sup>nd</sup> – 3<sup>rd</sup> May 2012
12. National Conference on Functional Nanomaterials: Synthesis, Characterization and Applications, Department of Physics, University of Pune during 31<sup>st</sup> Jan – 2<sup>nd</sup> Feb, 2013
13. UGC Sponsored National Seminar on “ Developments in Thin Film Processing & Characterization Technology” at BVDU, Yashwantrao Mohite College, Pune during 8<sup>th</sup> – 9<sup>th</sup> Oct, 2012
14. National conference on semiconductor materials and technology (NCSMT), Gurukula Kangri Vishwavidyalaya, Haridwara. Oct.16-18, 2008
15. International conference on Nanotechnology -2012 at Faculty of Industrial Science and Technology, University Malaysia Pahang during 30<sup>th</sup> May-1<sup>st</sup> June 2012
16. Third Science Conclave: A Congregation of Nobel Laureates, an MHRD & DST initiative, organized by Indian Institute of Information Technology (IIIT), Allahabad, during 8<sup>th</sup> to 14<sup>th</sup> Dec, 2010
17. An “Awareness Program on JCCC@UGC INFONET” organized by Bharati Vidyapeeth Dental College and Hospital, Pune on 19<sup>th</sup> Dec, 2008
18. Research invited visit to Korean Institute of Science and Technology (KIST), Seoul, South Korea with co-guide Prof. R. S. Mane during 1<sup>st</sup> Oct-30<sup>th</sup> Dec, 2009

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