Synthesis and characterization of pure and doped ZnO thin films by colloidal solution route

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Table of Content

Title of the thesis and abstract:

Title: Synthesis and characterization of pure and doped ZnO thin films by colloidal solution route

Abstract:

ZnO, a II-VI group semiconductor having a direct wide band gap (3.37 eV) and large exciton binding energy (60 meV), has great attention of researchers because of its electrical and optical properties which makes it an important material in various applications. ZnO thin film exhibits hexagonal wurtzite crystal structure. High excitonic binding energy, high resistivity against radiation, high breakdown voltage, sensitivity to visible light, and easy wet chemical stability are some of the interesting features of this material. ZnO is mostly being deposited using two ways: vacuum deposition and chemical route. The main drawback of vacuum deposition is the requirement of expensive vacuum equipments. Additionally the throughput of this method is less compared to chemical (colloidal) one. The present work focuses the Chemical Bath Deposition (CBD) method due to its simplicity in requirements. Another advantage of the CBD method over other methods is that the film can be deposited on substrates of different shapes and size. Influence of multiple layering of pure ZnO thin films on the structural, morphological and optical properties are studied. The films of various thicknesses and particle size are prepared by varying the concentration of $ZnCl_2$ as precursor material from 0.1M to 0.5M. Structural, morphological, optical and electrical properties of pure zinc oxide (ZnO) thin films grown by CBD and dip coating method on glass substrates and effect of doping various materials such as copper (Cu: ZnO), sodium (Na: ZnO) and potassium (K: ZnO) on the properties of ZnO thin films grown by CBD on glass substrates are investigated.

We can summarize the work done in present thesis in the form of different chapters.

Chapter ONE deals with the general introduction of the subject matter of the thesis.

Chapter TWO included a brief discussion of the literature review and aim of the work.

Chapter THREE deals with instrumental techniques and theoretical considerations.

Chapter FOUR discusses the preparation of ZnO thin films by CBD and their characterizations

Chapter FIVE discusses the preparation of ZnO thin films by dip coating, powder sample and their characterization

Chapter SIX discusses the preparation of doped ZnO thin films (Cu: ZnO, K: ZnO and Na: ZnO) by CBD and their characterizations

Chapter SEVEN included a summary, conclusions and scope of future work

Brief description on the state of the art of the research topic:

ZnO belongs to the II-VI compound semiconductors having n-type conductivity and 3.37 eV band gap [1]. This wide direct band gap makes it potentially important material for several applications such as solar cell, chemical sensors, electroluminescent devices and ultraviolet laser diodes[2-3]. It is also attractive in the field of semiconductor due to its excellent optical properties, high stability, cheap and abundant element and good electrical properties compared to other materials [4].

The chemical bath deposition technique is simple, cost effective, and reproducible. As compared to other oxide material ZnO material is much cheap and raw materials required for it are easily available. Several researchers have used CBD to deposit pure [5] and doped [6] ZnO thin films and to control its structural, morphological and optical properties by studying the influence of variation in concentration [7] and number of coat[8] of precursors.

ZnO have been studied as the active channel material in thin film transistors development because of its n-type nature, very good thermal stability and ability to crystalline orientation on different substrates [9]. Hence, the focus is to synthesize pure ZnO and copper (Cu), potassium (K) and sodium (Na) doped ZnO thin films on glass substrate by colloidal solution route and to study effect of doping on the electrical and optical properties of the thin films.

The n-type ZnO is available even without any intentional doping, but it is very difficult to get p-type ZnO. Many groups have realized p-type ZnO conversion by doping group V elements (N [10], P [11], As [12], and Sb [13] and groups I and IB (Li [14], Na [15-16-17], K[18], Ag[19], and Cu [20]), but the stability was not good. Reports of Na-doped p-type ZnO films are rather poor[21]. We report a simple, reliable, and low-cost method i.e. chemical route for growing p-type ZnO thin films in aqueous solution, with Cu, Na and K as the p-type dopant.

Problem Definition:

The variation in precursor concentration is one of the factors that plays a crucial role in the structural, morphological and optical properties of ZnO thin films. The literature survey shows that the effect of variation in number of coats i.e. from single coating to quintuple coatings and variation in precursor concentration i.e. from 0.1M to 0.5M need further studies.

As per the literature, comparative study on the various properties of pure ZnO and Cu, Na as well as K doped ZnO thin films along with variation in precursor concentration has not been reported yet. Doping asymmetry problem in ZnO in which n-type doping is easily achieved while p-type is quite problematic in terms of stability is well known which is a major hurdle for its application in optoelectronics and thus widespread development of ZnO-based devices has been inhibited. ZnO is an n-type semiconductor. ZnO is expected to be p-doped by elements of IA group (alkali metals such Li, Na, and K), IB group (transition metals such as Cu, Ag, Au), or V–A group (such as N, P, As, Sb) and still research is going on. Also various properties of ZnO thin films doped with transition metal and alkali metal have not been compared. Improvement in structural defects can be reduced using variation in number of coatings and variation in precursor concentration.

Objective and Plan of work:

Considering the present scenario of ZnO research, few issues were identified which have been the motivation of present thesis work. ZnO; both pure and doped has a lot of practical applications in different devices. The aim of this work is to offer some understanding of the variation in precursor concentration, variation in no. of coat as well as effect of doping on ZnO thin films.

ZnO thin films were used as a substrate to deposit another thin film of ZnO on them. Total 5 multilayer thin films were made by depositing ZnO thin films one by one and marked as 1C, 2C, 3C, 4C and 5C for Single coat, Double Coat, Triple Coat, Quadruple coat and Quintuple coat ZnO thin films respectively. We also report properties of ZnO thin films deposited by bath consisted of equal volumes of zinc chloride ($ZnCl_2$) solutions having concentrations varying from 0.1 M to 0.5 M. The experimental results show that the varying precursor concentration of the reactants has a remarkable and significant effect on the growth and characteristics properties of ZnO thin films.

Many investigations are carried out to improve the characteristics of ZnO thin films by doping in it with various materials. However, investigations of Cu, Na and K doped ZnO thin films are very scarce, also to the best of our knowledge, there is no study which compares the effects of these dopants on electrical and optical properties of ZnO. Therefore, we set out an easy, well-grounded and low-cost method; Chemical Bath Deposition in aqueous solution for growing and doping ZnO with Cu, K and Na dopant. Influence of doping on electrical and optical parameters like resistivity, conductivity, mobility, carrier concentration, band gap values and ionization energies of impurity levels were investigated.

Precisely the objectives are given below,

1) To prepare ZnO both pure and doped through an easy and cost effective method which can be made commercially viable.
2) Transmittance, absorbance, optical absorption coefficient, optical band gap (E_g), refractive index, dielectric constants, etc. would be determined from UV-visible spectra.
3) Proper characterization like thermos-gravimetric Analysis (TGA), Scanning Electrons Microscope (SEM), X-Ray Diffraction (XRD), Photoluminescence, RAMAN Spectroscopy, Resistivity Measurement, Hall Effect, Thermoelectric power, Peltier coefficient and power factor of these pure and doped ZnO thin films to resolve different issues.
4) Effect of doping on the various properties of ZnO thin films.
5) To establish a structural, optical and electrical property correlation for better understanding.
6) Direction for further research work in this area in future.

Original contribution by the thesis:

Zinc oxide thin films were deposited on glass substrate from aqueous solution of $ZnCl_2$ and NH_3 by chemical bath deposition method (CBD). Films of various thicknesses, particle size and other useful analysis have been obtained by varying the concentration of $ZnCl_2$ from 0.1M, 0.2M, 0.3M, 0.4M and 0.5M. Optical properties of grown films and influence of precursor concentration on them are analyzed by the transmittance recorded in the range 200-1200 nm. Deposited films are also characterized by powder X-ray diffraction and SEM to understand the influence of variation in precursor concentration on structural and morphological properties of grown films. XRD patterns confirmed the hexagonal wurtzite structure of the deposited pure ZnO films.

Thin films having different number of layers were prepared by depositing ZnO layers on one after other. Influence of multiple layering of pure ZnO thin films on the structural, morphological and optical properties are studied by X-ray diffraction, scanning electron microscopy and UV-VIS-NIR transmission spectroscopy. Required annealing temperature for the as grown films is determined by TGA analysis of powder obtained by drying precursor solution.

The effects of copper (Cu) dopant on the properties of ZnO thin films grown by chemical bath deposition (CBD) and dip coating method on glass substrates are investigated. Optical properties are investigated through UV-VIS-NIR transmittance and reflectance spectroscopy. Photoluminescence spectra of the samples show prominent peaks corresponding to blue emission and defect related green emission in the visible region at room temperature and their possible mechanism have also been discussed. Hall Effect Measurements, Resistivity, Raman

and photoluminescence (PL) studies of pure ZnO and copper (Cu), potassium (K) and sodium (Na) doped ZnO thin films on glass substrate by colloidal solution route have been performed. The influence of dopant content on carrier concentration, electrical resistivity, and hall mobility of the thin films were analyzed from Hall Effect measurements. Temperature dependent electrical resistivity measurements are carried out by two probe method and activation energy for the electrical conductivity of pure and doped ZnO are determined. The Raman scattering of the pure ZnO and Copper (Cu), Potassium (K) and Sodium (Na) doped ZnO shows the first and second orders of polar and non-polar modes. Photoluminescence (PL) at room temperature results indicate the emission occurs at close band lines and the outcomes are identified with a few inherent imperfections in the doped ZnO thin films. Raman spectroscopy and photoluminescence confirm existence of zinc interstitials (Zni) as well as oxygen vacancies (V_o). The spectroscopy analysis to the samples resulted the importance of phonon-electron connections, assuming the essential role play in the semiconductor e.g. ZnO.

Methodology of Research and Results:

The research focused on the synthesis and characterization of pure zinc oxide, copper doped ZnO (Cu: ZnO), sodium doped ZnO (Na: ZnO) and potassium doped ZnO (K: ZnO) thin films aims the preparation of the thin films by colloidal solution route.

Effects of multi-layering and increasing in precursor concentration from 0.1M to 0.5M of ZnO thin films on morphology and optical properties were studied. Lattice strain, band gap, refractive index and extinction coefficient are found to be firmly unaffected by multiple layering of ZnO thin films, while surface morphology, crystallite size, transmittance and absorbance changes with number of coats.

Thermal behavior of dried powder of precursor complex was analyzed by thermo-gravimetric analysis (TGA) (Fig.1) and differential thermal analysis.

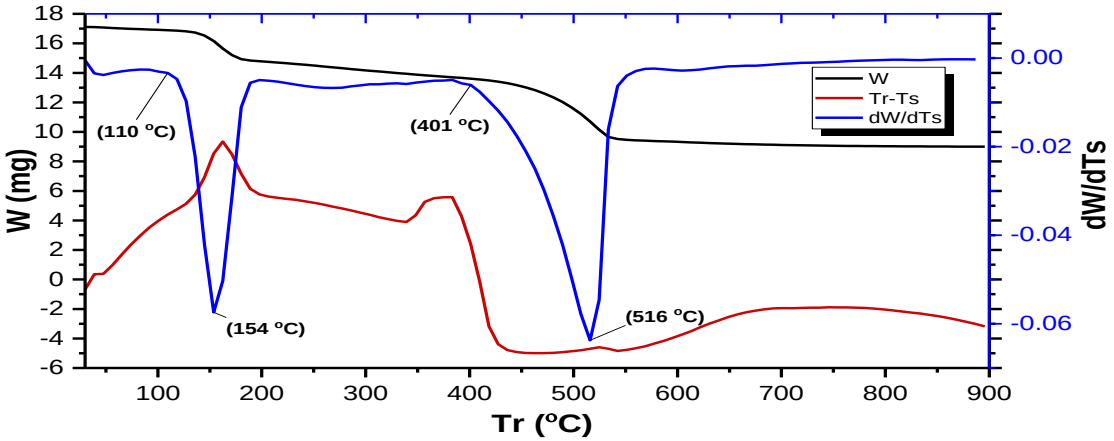


Fig. 1 TGA-DTA curve of dried precursor complex used synthesize ZnO thin films.

The result of above curve shows that annealing temperature required for the conversion of precursor complexes into ZnO is 518 ◦C.

Surface morphology of thin films was studied by scanning electron microscope (SEM) (Fig.2). ZnO thin films are found to be grown in the form of randomly oriented hexagonal pillars having micrometer sized diameter. Size of hexagonal pillars are found to be increasing, also these surface structures seem to be agglomerating with increasing number of coat as well as precursor concentration. After certain point even though the surface is porous nucleation is found to be taking place on the top of the hexagons followed by the growth of new random oriented hexagonal pillars having size comparable to the size of initial pillars. Cross-sectional SEM micrographs indicate the increase in thickness of ZnO thin film with increase in number of coat as well as precursor concentration. Average size of hexagonal surface structures and thickness of ZnO thin films are measured for multilayer thin films and variation in precursor concentration from 0.1M to 0.5M.

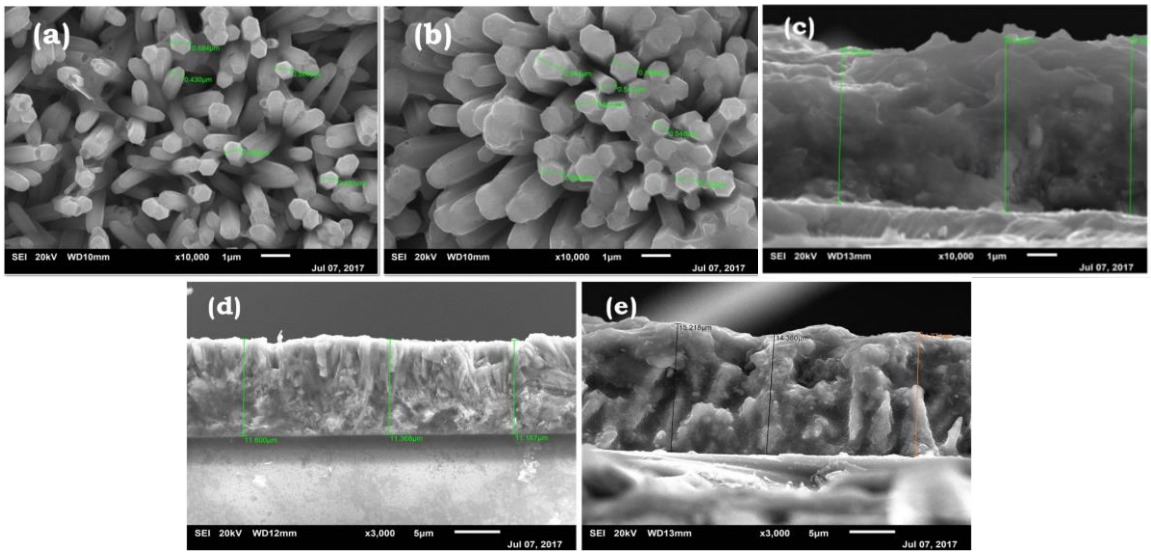


Fig.2 SEM micrographs and cross-sectional SEM micrographs of ZnO multilayer thin films

Structural characterization of the thin films was performed by X-ray diffraction. Diffraction peaks were compared with those of the standard compounds reported in the JCPDS files.

The crystallite size and band gap energy were found to depend on the concentration while increasing concentration from 0.1M to 0.5M. The band gap increases with increase in the crystallite size. XRD data reveals peaks corresponding to various crystallographic planes i.e. (100), (002), (101), (102), (110), (103) and (200) and the films are found to be more oriented along (100), (002) and (101) directions as shown in figure 3.

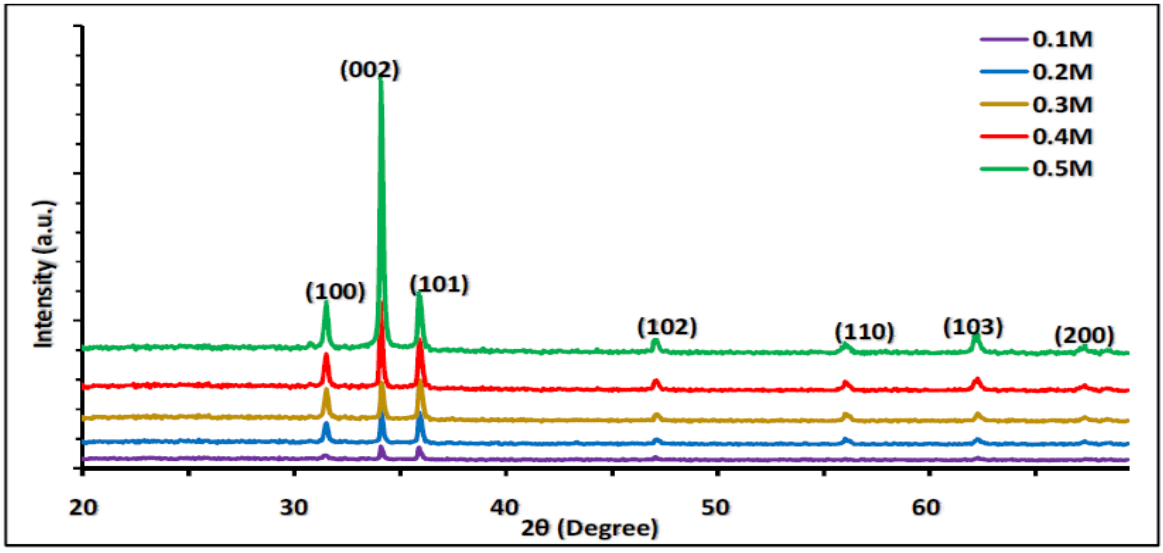


Fig. 3. Powder XRD patterns for variation in precursor concentration ZnO thin films.

UV-Vis-NIR absorption spectra of thin films were recorded in the wave length range of 200 nm to 1200 nm. As the concentration of the precursor increases, thickness of the film is increasing and lattice strain is also increasing gradually. Transmittance spectra shows that transparency of the films ranges from 33 to 54%.

Band gap energy (Eg) was determined by plotting $(\alpha h\upsilon)^2$ vs $h\upsilon$ and extrapolating the linear portion of the plot to the X-axis (Fig. 4 and 5).

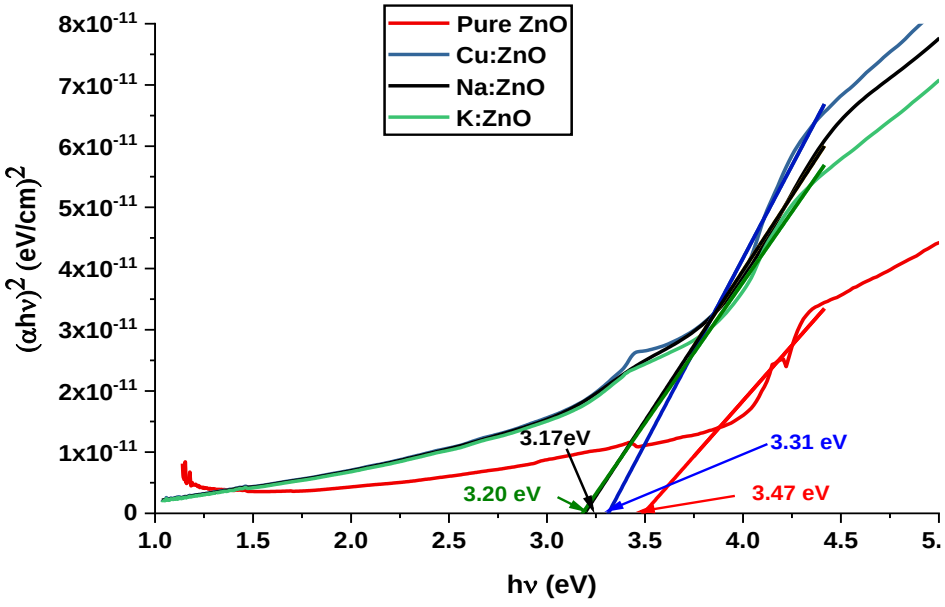


Fig.4 $(\alpha h\nu)^2$ → photon energy of pure ZnO, Cu:ZnO, Na:ZnO and K:ZnO thin films grown using CBD.

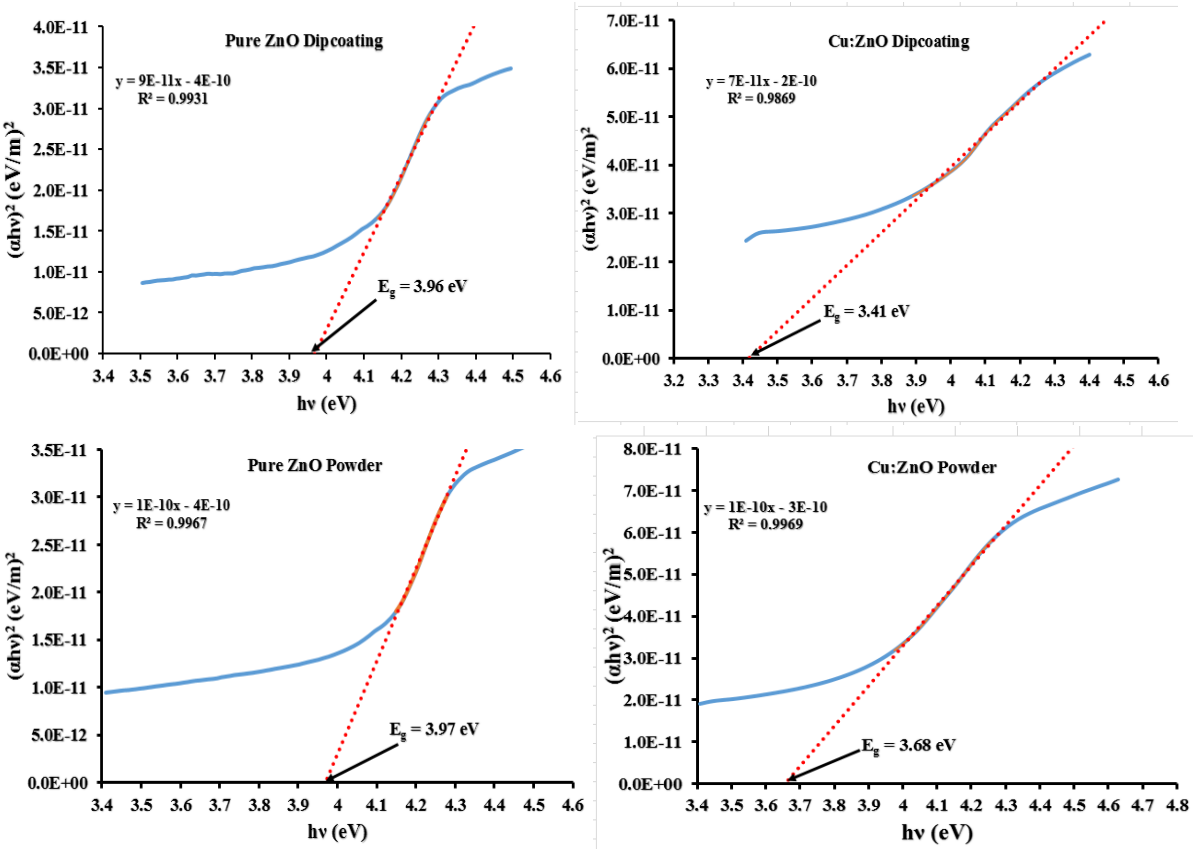


Fig.5 $(\alpha h\nu)^2$ → photon energy of pure ZnO, Cu:ZnO, Na:ZnO and K:ZnO thin films grown using Dip coating.

A reduction in band gap value is observed with doping. Systematic decrease in band gap could not be due to structural changes by adding dopant into ZnO. Narrowing of band gap is due to effects on conduction and valence bands [22] which can shrink band gap originate from electron interaction and impurity scattering. It has been attributed to merging of an impurity band into conduction band, thereby shrinking band gap. A widening of the band-gap indicates ZnO doping with donors; while p-type ZnO has indicates reduced band-gap whenever doped with acceptors. By increasing the precursor concentration from 0.1 to 0.5 M of Cu, Na and K doped ZnO, the resistivity value decreases.

Raman Microscope with a laser excitation source was used to record Raman scattering spectra at room temperature. Raman spectra in figure 6 shows that some of the additional peaks appear in the spectra of ZnO thin films with certain dopant species, indicating host lattice defects in the ZnO structure only.

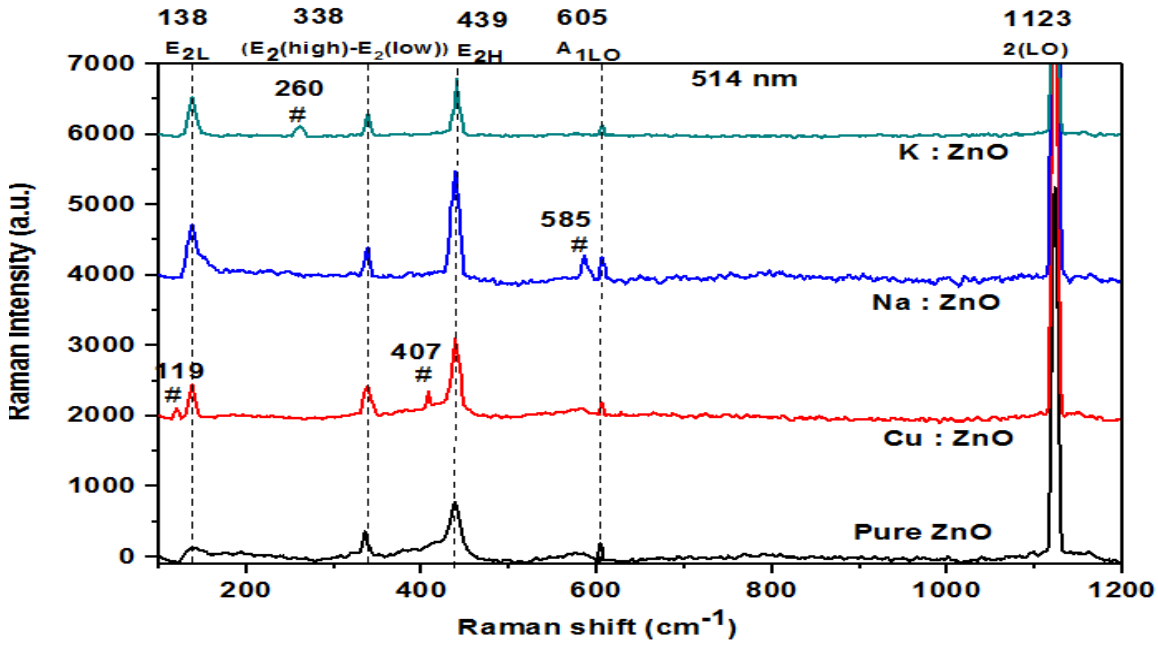


Fig.6 Raman spectra of undoped and doped ZnO films grown on glass substrate.

PL (photoluminescence) spectrum of all the samples were recorded at room temperature. The red-shift in UV emission is observed in PL spectra of doped ZnO. This red-shift in the UV emission for doped ZnO could be an outcome to get p-type ZnO.

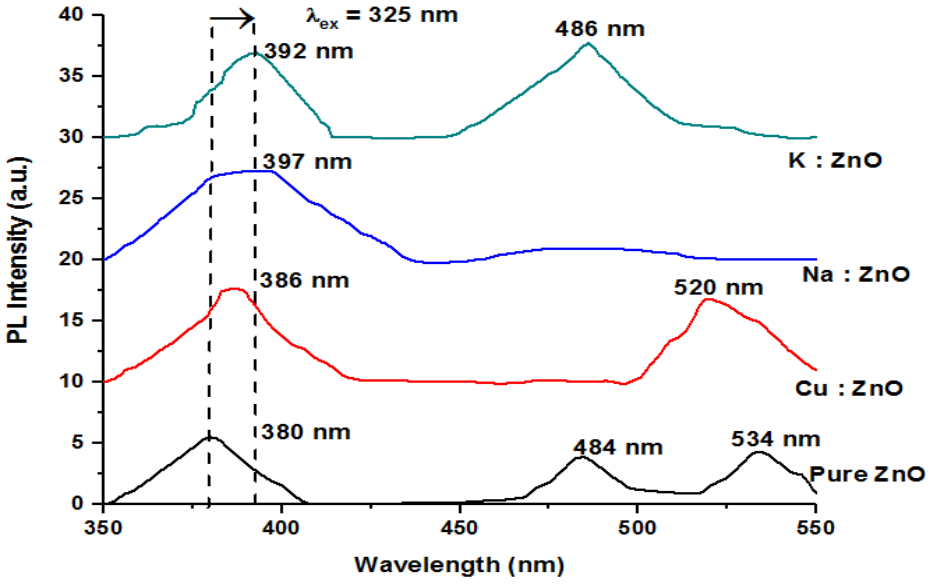


Fig. 7 Red-shift of the UV peaks in PL spectra for the Cu-, Na- and K- doped ZnO compared to the undoped ZnO thin films.

The variation of log ρ with reciprocal of temperature for the Cu, Na and K doped ZnO thin films (Figure 8). It is observed that resistance increases with increase in temperature indicating semiconducting nature of the films. From the slopes of log ρ vs 1000/T plots the values of activation energies were calculated.

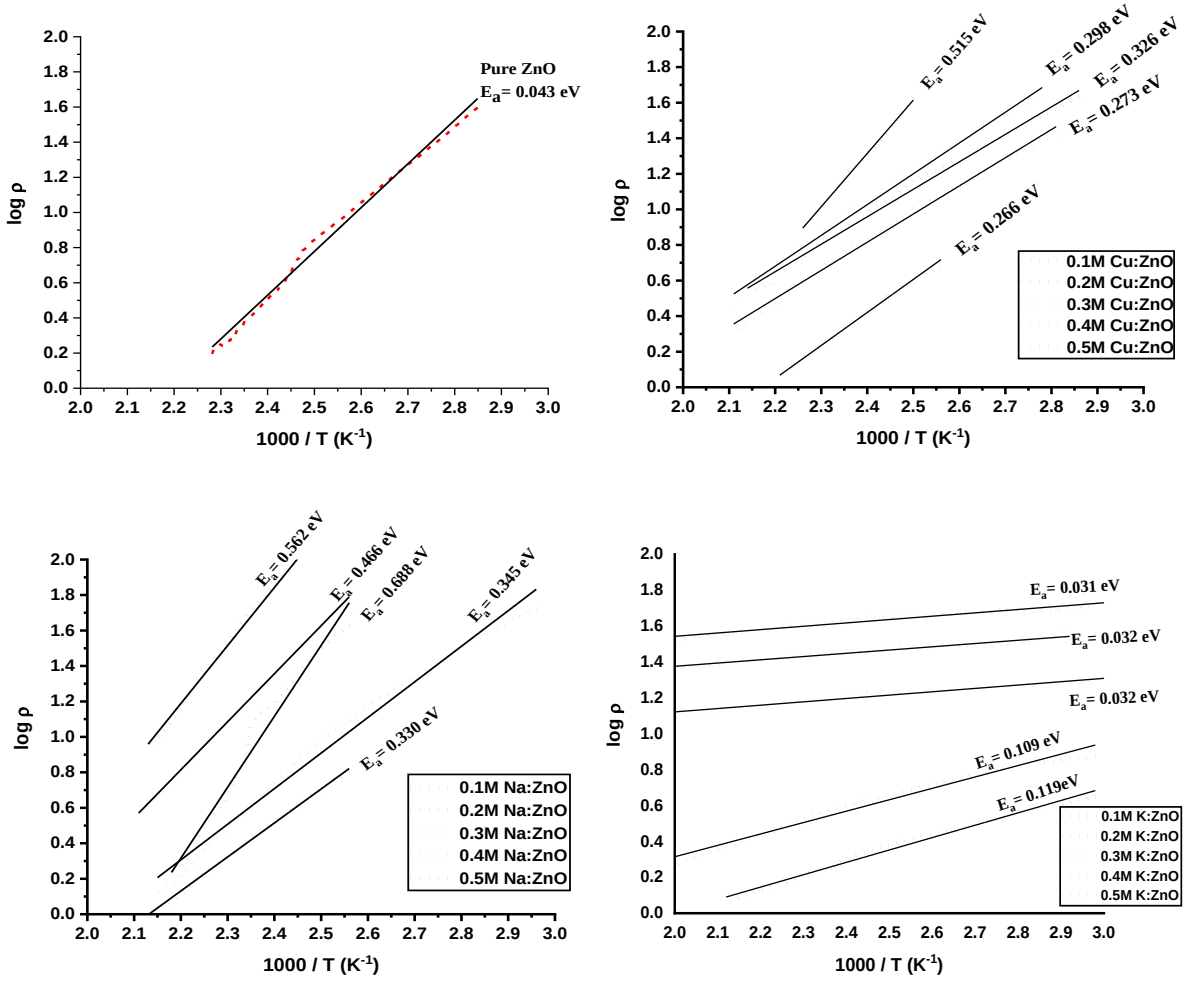
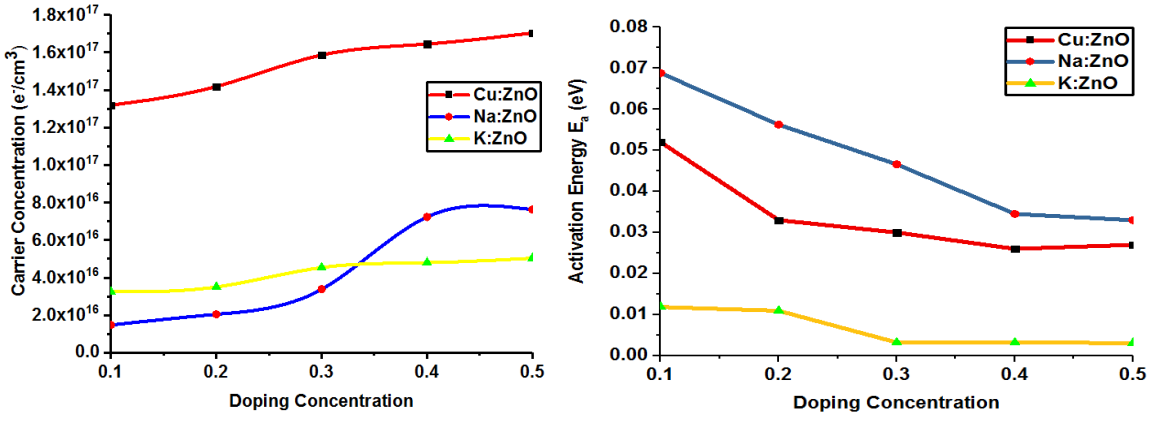


Fig.8 log ρ vs 1000/T for pure and Cu, Na and K doped ZnO thin films.

Hall-effect measurements were carried out in the van der Pauw configuration at room temperature for the determination of the type of conduction mechanism, Hall mobility and carrier concentration. Electrical conductivity and activation energy were determined by the two probe resistivity measurement performed on all the samples from room temperature to 200 ˚C as shown in figure 9.



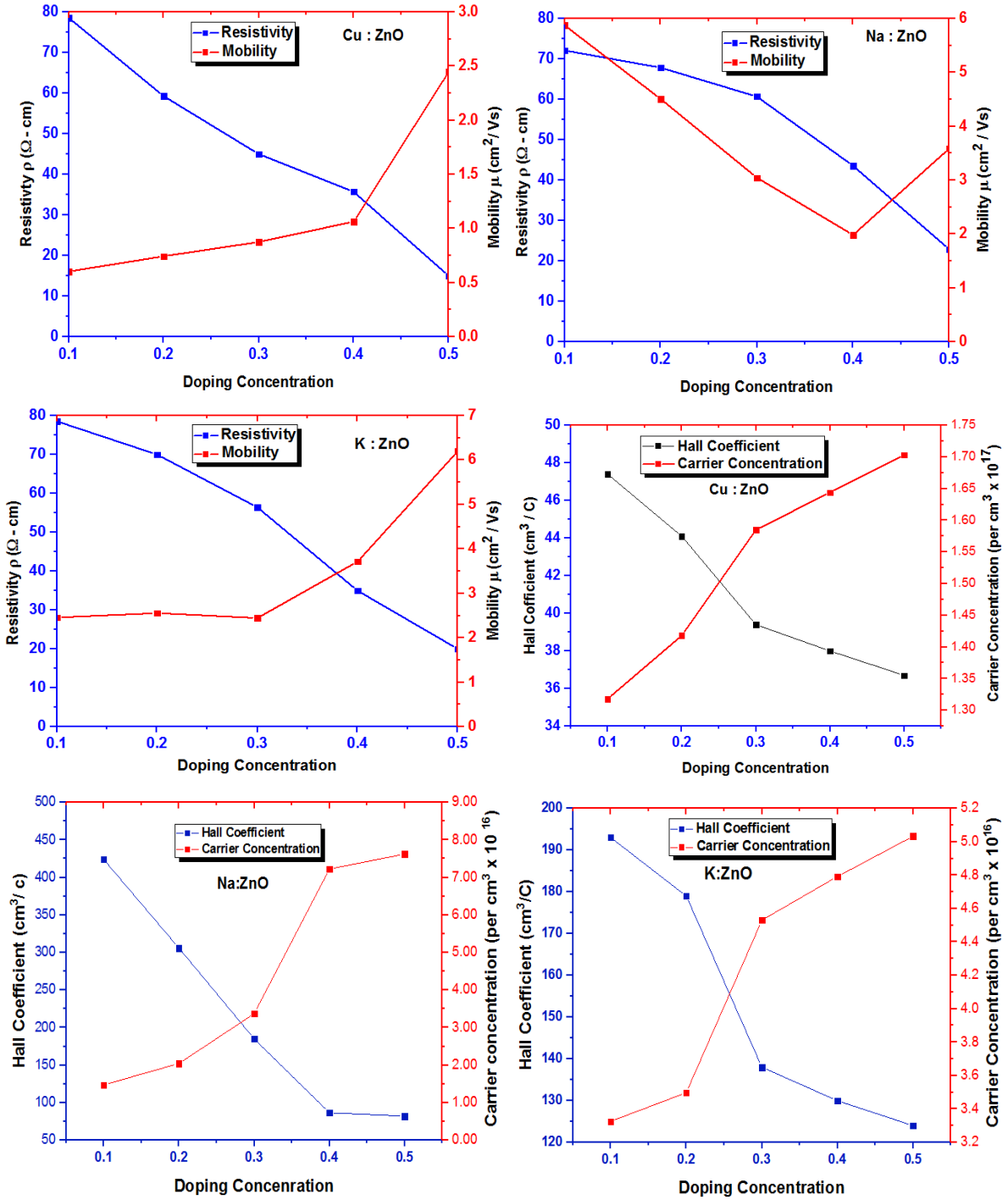


Fig. 9. Resistivity, carrier concentration, mobility hall coefficient and activation energy of a Cu, Na and K doped ZnO thin films vs doping concentration

The positive value of Hall voltage of all doped thin films measured using Vander Pauw method with four point electrode fixture is in agreement with the red shift in PL (Fig.7) spectra of p-type doping.

Complete and careful comparison of pure ZnO and copper (Cu), potassium (K) and sodium (Na) doped ZnO thin films has been carried out. All three dopants improve optical and electrical properties of films compared to undoped ZnO.

The thermoelectric power has been measured from room temperature up to 200 ^{0}C for all sixteen samples containing pure ZnO and Cu, Na and K doped ZnO thin films which are grown on glass surface by CBD route with variation in precursor concentration from 0.1M to 0.5M.

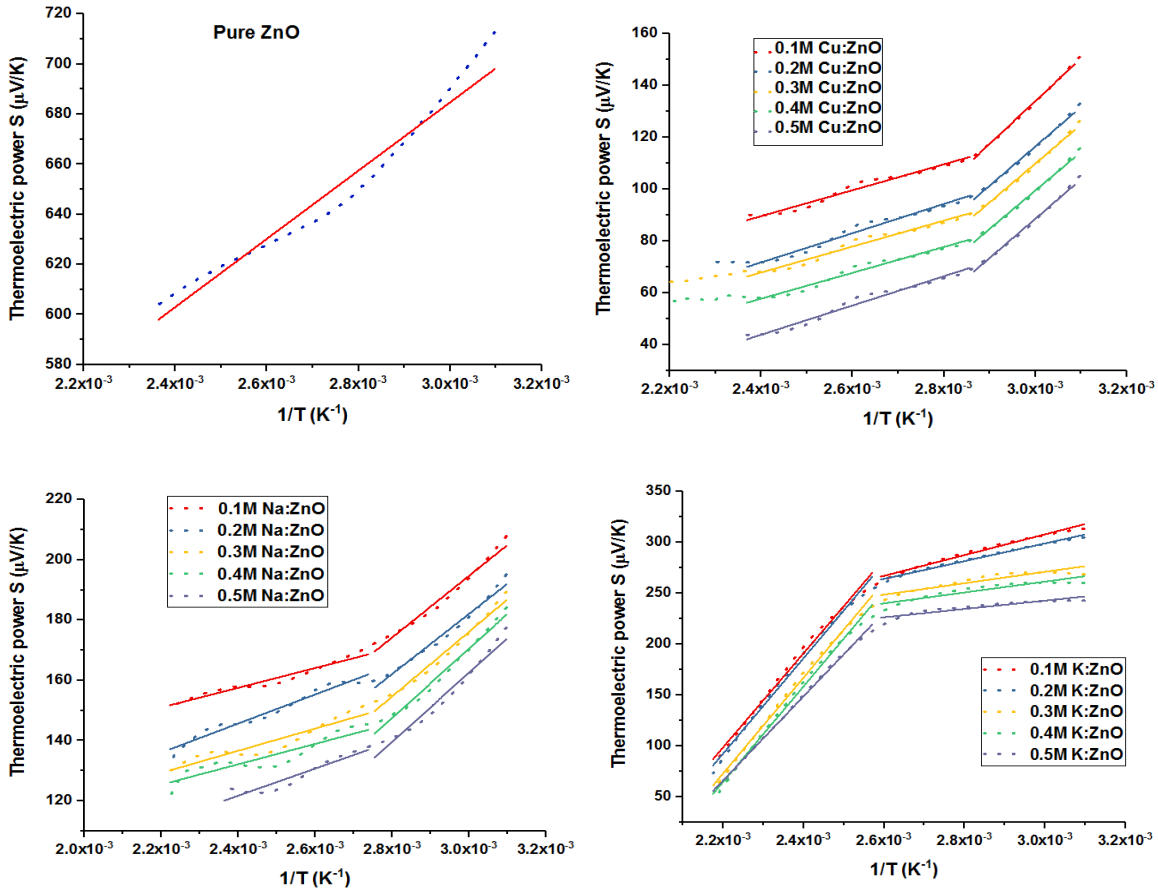


Fig.10 Thermoelectric power vs inverse temperature for pure ZnO, Cu: ZnO, Na: ZnO and K: ZnO thin films

It is observed from figure 10 that the thermo power decreases continuously with increase in temperature. The curves give an intercept at the ordinate from which the value of A has been obtained. This value corresponds to the scattering index and is an indication that piezoelectric scattering is dominant in these ZnO films. This is in agreement with the fact that ZnO is a piezoelectric crystal. Also charged as well as neutral impurity scattering in Cu and optical mode, piezoelectric scattering and acoustic phonon scattering in K doped ZnO thin films grown using CBD method with the variation in concentration from 0.1M to 0.5M at different temperature is observed.

Peltier coefficient is also calculated which is the energy carried by the electrons per unit charge. The equation of Peltier coefficient (π) shows that a π versus temperature plot should yield a straight line and the value of γ i.e. temperature coefficient can be obtained from its slope.

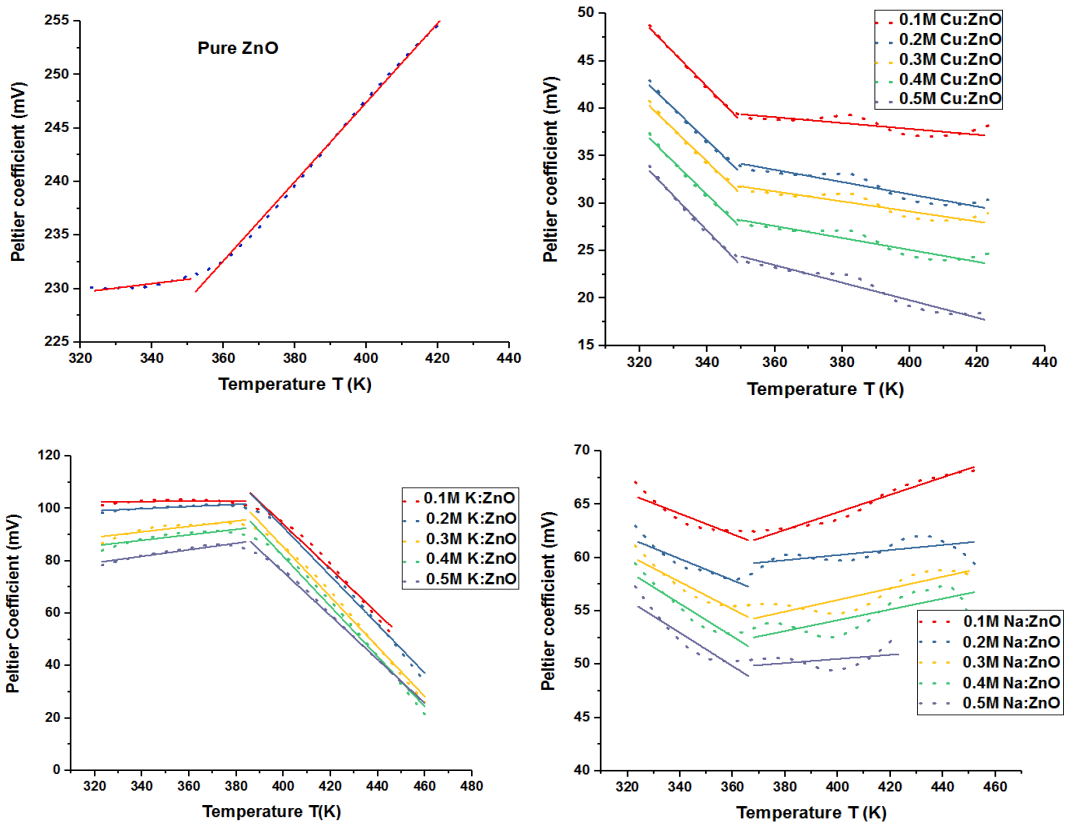


Fig.11 Peltier coefficient vs inverse temperature for pure ZnO, Cu, Na and K doped ZnO with variation in concentration from 0.1M to 0.5M.

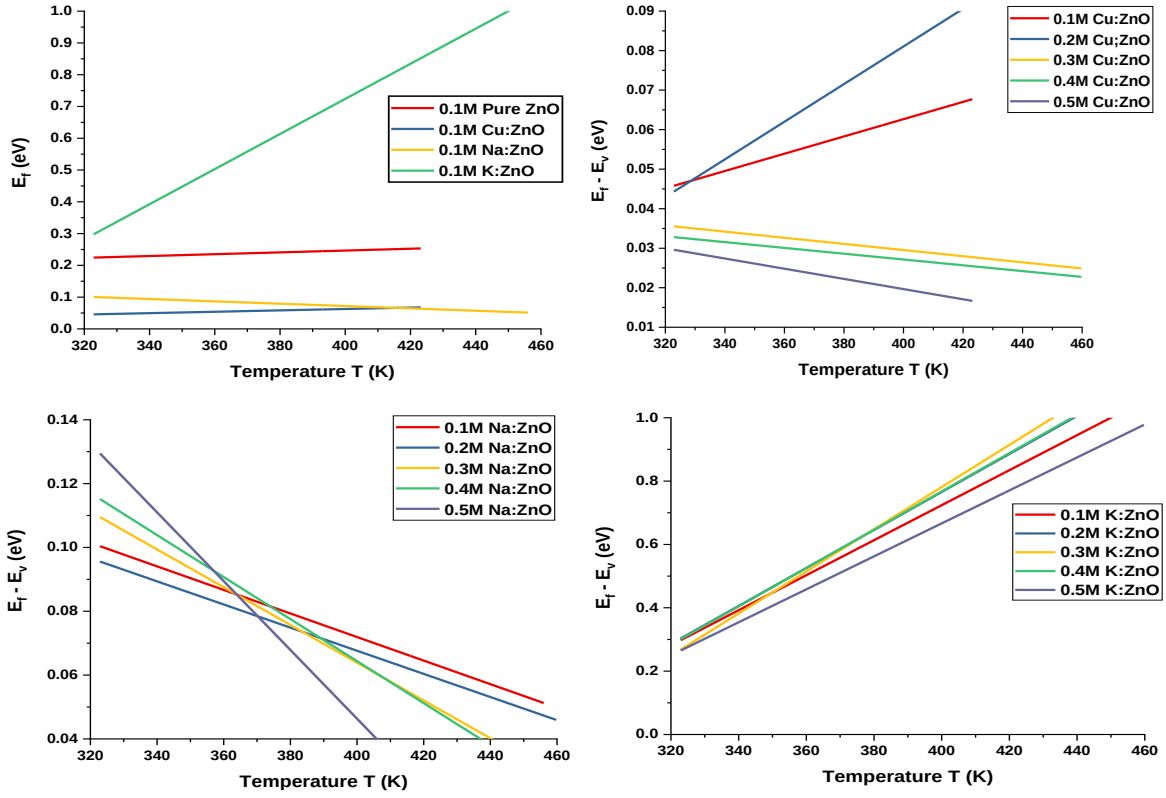


Fig.12 (E_c-E_f) vs temperature for Pure ZnO and (E_f-E_v) vs temperature for Cu, Na and K doped ZnO with variation in concentration from 0.1M to 0.5M.

An overview of the experimental results of the power factor (electrical conductivity times the square of Seebeck coefficient) for pure ZnO and Cu, Na and K doped ZnO thin films for the variation in precursor concentration from 0.1M to 0.5M is presented in figure 13. Power factor which determines the performance of the thermoelectric energy convertor.

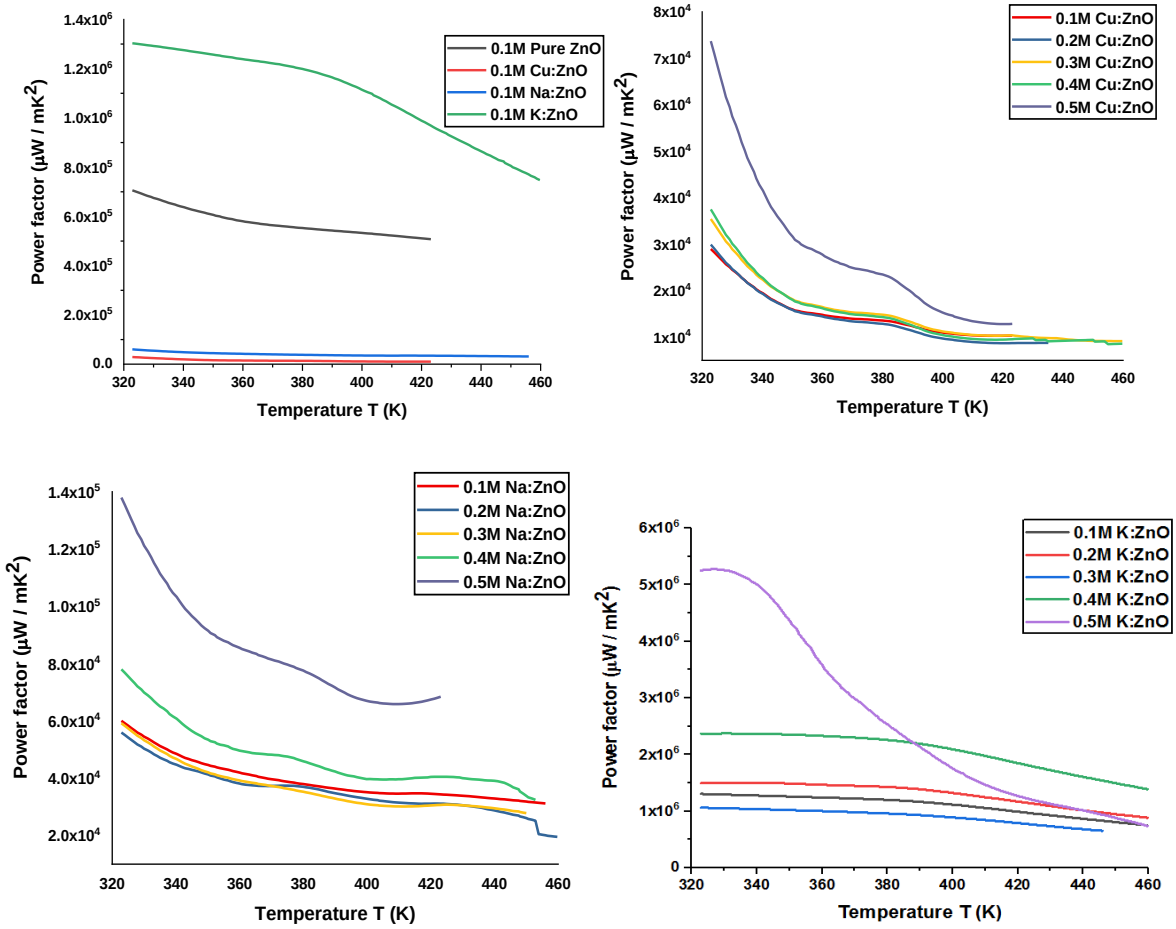


Fig. 13 Power factor vs temperature for pure ZnO, Cu, Na and K doped ZnO with variation in concentration from 0.1M to 0.5M.

Achievements with respect to objectives:

Thin films having different number of layers were prepared by depositing ZnO layers on one after other. Influence of multiple layering of ZnO thin films on the structural, morphological and optical properties were studied by X-ray diffraction, scanning electron microscopy and UV-VIS-NIR transmission spectroscopy.

The films of various thicknesses, particle size and other useful analysis have been obtained by varying the concentration of $ZnCl_2$ from 0.1M, 0.2M, 0.3M, 0.4M and 0.5M. As the concentration of the precursor increases, thickness of the film increases and lattice strain also

increases gradually. The crystallite size and band gap energy were found to depend on the concentration. The band gap increases as the crystallite size increases.

It is of interest to us to investigate the influence of the Cu, Na and K dopant elements on the properties of ZnO thin films deposited by chemical routes. Therefore, we set out an easy, well-grounded and low-cost method for growing and doping ZnO with Cu, K and Na dopant using Chemical Bath Deposition in aqueous solution. Influence of doping on electrical and optical parameters like resistivity, conductivity, mobility, carrier concentration, band gap values and ionization energies of impurity levels were investigated.

The positive value of Hall voltage of all doped thin films i.e. copper (Cu), potassium (K) and sodium (Na) doped ZnO thin films measured using Van der Pauw method with four point electrode fixture is in agreement with the red shift in PL spectra of p-type doping.

Seebeck coefficient, Peltier coefficient and power factor of pure and Cu, Na and K doped ZnO thin films grown using CBD method by varying precursor concentration from 0.1M to 0.5M are found, which show results in very positive manner.

Conclusion:

In the current research, pure ZnO, copper (Cu), potassium (K) and sodium (Na) doped ZnO thin films on glass substrate films as well as powder of ZnO have been prepared by colloidal solution route.

In pure ZnO thin films, the optical and electrical properties of ZnO thin films strongly varied with the increasing precursor concentrations from 0.1M to 0.5M as well as by varying number of coat from single coat to quintuple coat of ZnO thin films. All three dopants improve the optical and electrical properties of thin films compared to undoped ZnO. The Raman spectra shows that the intensity of the dominant peak changes with different dopant compared to the ZnO thin film. The shifting of peaks is due to lattice defects and lattice disorder. The synchronous Raman and photoluminescence examinations have the preferred standpoint to correspond with the changes observed in the optical property of the semiconducting materials. The positive value of Hall voltage of all doped thin films measured using Van der pauw method with four point electrode fixture is in agreement with the red shift in PL spectra of p-type doping. The decrease in resistivity of copper doped thin films compared with ZnO films is attributed to the replacement of Zn^{2+} ions by Cu^{2+} ions while in sodium and potassium doped thin films decrease in resistivity is due to the interstitial localization of Na^{+} and K^{+} ions respectively in the adjacent of the oxygen and Zn atoms. The resistivity of all doped thin films decrease with increase in doping concentration. Activation energy has been observed to decrease with increase in dopant concentration as well as change in dopant. ZnO thin films

doped with Cu, Na and K has the capability to increase its conductivity without adversely modifying other properties. Increase in doping material will reduce the Seebeck coefficient. Pure ZnO, 0.3M to 0.5M Na doped and all K doped ZnO thin films are nondegenerate, while all Cu doped and 0.1M to 0.2M Na doped ZnO are degenerate. Power factor of Cu, Na and K doped thin films increases as the variation in concentration increases from 0.1M to 0.5M. Power factor which determines the performance of the thermoelectric energy convertor. There is an optimum doping concentration for thermoelectric cooling or power generation applications.

List of Publications:

1. A paper title "Synthesis and Characterization of ZnO by Chemical Bath Deposition" published International Science Symposium on Recent Trends in Science and Technology Organized by Christ College, Rajkot & Sponsored by DST, Govt. of India, 2017.
2. A paper title "Effect of concentration of TEA and annealing temperature on band gap of ZnO: A review" published in International Journal of Research in Modern Engineering and Emerging Technology. 2017: 5(9), 100-106.
3. A paper title "Influence of Precursor Concentration on CBD grown ZnO Thin Films" published in International Journal of Scientific Research and Reviews. 2018: 7(1), 446-457.
4. A paper title "Influence of Multiple Layers on Chemical Bath Deposited ZnO Thin Films" published in International Journal for Research in Applied Science & Engineering Technology. 2018: 6(3), 268-274.
5. A paper title "Growth, synthesis, structural and optical properties of pure ZnO by colloidal solution route" published in Journal of Emerging Technologies and Innovative Research 2019: 6(1), 693-699.
6. A paper title "Effect of doping concentration on optical and electrical properties of intrinsic n-type ZnO (i-ZnO) and (Cu, Na and K) doped p-type ZnO thin films grown by chemical bath deposition method" published in Nanosystems Physics, Chemistry, Mathematics, 2020, 11 (4), 391–400.
7. A paper title "Synthesis, optical and photoluminescence properties of undoped and Cu-Doped ZnO thin films by colloidal solution route" published in Molecular Crystals and Liquid Crystals, 2020, 712 (1), 62-75.

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