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PERFORMANCE EVALUATION OF ZNO DOPED DIDYMIUM NANO POWDER SAMPLES

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Abstract- In the recent years, much attention has been focused on wide band gap semiconductors materials because of their excellent potential for blue light emitting devices, short-wavelength laser diodes and detectors in UV-blue spectral region. The wide band gap ZnO is gaining much importance for the possible application due to the capability of ultraviolet lasing at room temperature and possibilities to engineer the band gap for further use. In order to attain the potential offered by ZnO, both high-quality n-and p-type ZnO are essential. In this work we synthese the ZnO nanopowder by Sol–gel method & after that the ZnO is doped with rare earth material didymium. Didymium is a naturally occurring element with major constituent Nd and Pr and is used in many applications. The FTIR, SEM and EDX characterization techniques are applied to study the sample & it has been found that the formation of pure phase of ZnO having wurtzite hexagonal structure occurs at 1%, But at higher concentration of Di (3%, 5%) the system shows mixed phase. Moreover SEM shows that the Di doped ZnO has well ordered morphology, has low aggregation and homogeneous distribution of particle size. Note that the synthesized system is also having band gap of 3.24 eV which is comparable with the standard value.

Keywords- *Rare Earth, Sol-gel, Doping, SEM, FTIR*

I. INTRODUCTION

Zinc oxide (ZnO) is a piezoelectric, dielectric, transparent, semiconducting oxide, with a direct band gap of 3.37 eV at room temperature and a large excitation binding energy (60 meV), which is 2.4 times the effective thermal energy (KBT=25meV) at room temperature, and bi excitation energy is 15meV. This is one of the key parameters that ZnO exhibits near-UV emission, transparency, conductivity, and resistance to high temperature electronic degradation. In addition, ZnO is the hardest of the II-VI semiconductors due to the higher melting point (2248k) and large cohesive energy (1.89ev) (therefore more resistant to wear), as well as one of the most piezoelectric semiconductors (d= 12.2 × 10-12 C/N) with good piezoelectric coefficient KL =0.27 and its high adherence on various substrates[1-3]. In the recent years, much attention has been focused on wide band gap semiconductors materials because of their excellent potential for blue light emitting devices, short-wavelength laser diodes and detectors in UV-blue spectral region.

As wide band gap ZnO is gaining much importance for the possible application due to the capability of ultraviolet lasing at room temperature and possibilities to engineer the band gap. In order to attain the potential offered by ZnO, both high-quality n-and p-type ZnO are essential. But it is very difficult to obtain the bipolar carrier doping (both n and p types) in wide-band-gap semiconductors such as GaN and II-VI compound semiconductors including ZnS, ZnSe, and ZnTe [4,5]. Unipolar doping has not been a surprising issue in wide-band-gap semiconductors: ZnO, GaN, ZnS, and ZnSe are easily doped to n-type , while p-type doping is difficult. All undoped ZnO to date has been found to be n-type, with donor concentrations typically around 10^{17} cm^{-3} for present-day, high-quality material, but sometimes as high doped material. The situation is opposite for ZnTe where p-type doping is easily obtained, while n-type doping is difficult. The main characterization techniques[5,6] used to find the shallow electrical defects in semiconductor materials are photoluminescence and temperature dependent Hall Effect measurements.In electronics world ZnO has played an important role in the fabrication of transparent thin film transistors (TFT), by depositing channel layer on a flexible substrate through low temperature processes, realizing transparent TFTs, and achieving extra functions such as photo detections using ZnO channel. In this case the protective covering to prevent light exposure is eliminated since ZnO based transistors are insensitive to visible light. The deposited ZnO usually maintains a crystalline phase, although the deposition process is carried out even at room temperature [6, 7].

In this work we synthesis the ZnO nanopowder by Sol –gel [11] method & after that the ZnO is doped with rare earth material didymium. We know that Didymium is a naturally occurring element with major constituent Nd and Pr. The FTIR, SEM and EDX characterization techniques are applied to study the samples & it has been found that the formation of pure phase of ZnO having wurtzite hexagonal structure. But at higher concentration of Di (3%, 5%) the system shows mixed phase. Moreover SEM shows that the Di doped ZnO has well ordered morphology, has low aggregation and homogeneous distribution of particle size Note that the synthesized

system is also having band gap of 3.24 eV which is comparable with the standard value.

II. ZINC OXIDE AND DIDYMIUM

ZnO is a II-VI semiconductor with wide directgap(3.37 eV) and exciton binding energy (60 meV)at room temperature. It is an inexpensive and environmentally safe host material. Due to its properties, the interest in ZnO as a photocatalyst has increased, however it has been mainly used under ultraviolet (UV) irradiation [8-10].Actually ZnO was one of the first semiconductors to be prepared in rather pure form after silicon and germanium. It was extensively characterized as early as the 1950's and 1960's due to its promising piezoelectric/acousto electric properties. Wide band gap semiconductors have gained much attention during last decade because of their possible uses as optoelectronic devices in the short wavelength and ultraviolet (UV) portion of the electromagnetic spectrum. Zinc oxide[12,13] crystallizes in three forms: hexagonal wurtzite, cubic zinc blende, and the rarely observed cubic rocksalt). The wurtzite structure is most stable and under ambient conditions the wurtzite phase is energetically favorable. This structure is composed of two interpenetrating hexagonal closed packed sublatices of cation (Zn2+) and anion(O2−) stapled ABAB. . . along the c axis as shown. In Wurtzite crystal structure of ZnO each atom is bound tetrahedrally to four atoms of the other kind as ahown in figure 1. Note that the other two structures i.e. cubic zinc blende, and cubic rocksalt are rarely realized since they need either very high pressure or uncommon substrates.

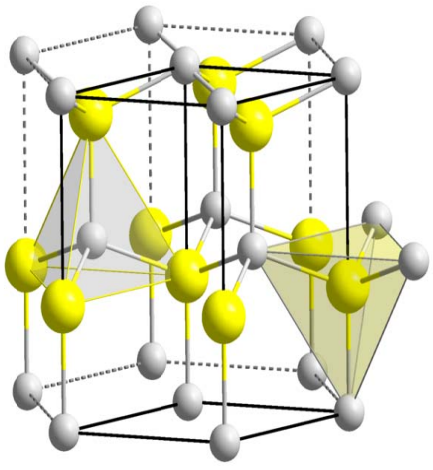

Figure 1. Wurtzite crystal structure of ZnO

Didymium [14,15] (Greek: twin element) is a mixture of the elements praseodymium and neodymium. Didymium was discovered by Carl Mosander in 1841 and was so named because it is very similar to lanthanum, with which it was found It is used in safety glasses for glassblowing and blacksmithing, especially when a gas (propane) powered forge is used, where it provides a filter which selectively blocks the yellowish light at 589 nm emitted by the hot sodium in the glass, without having a detrimental effect on general vision, unlike dark welder's glasses. Blocked also is the strong ultraviolet light emitted by the superheated forge gases and insulation lining the forge walls thereby saving the crafters' eyes from serious cumulative damage. Didymium photographic filters are often used to enhance fall scenery by making leaves appear more vibrant. It does this by removing part of the orange region of the color spectrum, acting as an optical band-stop filter. Unfiltered, this group of colors tends to make certain elements of a picture appear "muddy". The "Sodium Vapor Process"[16] used in motion picture matte work included a didymium filtering prism in the camera. Didymium is also used in calibration materials for spectroscopy.

III. SYNTHESIS OF ZNO NANO POWDER SAMPLE

In this process the glass wares (three necks round bottom flask, measuring cylinder, beaker) is first cleaned and rinse with distilled water and dried in vacuum oven. All the materials and solvents are weighted with help of electronic weighing balance and mixed in cleaned round bottom flask. A 100 ml three neck flask charged with Zinc acetate and ethanol. The surfactant Didymium is doped at different concentration. All the reactions are refluxed at 74^{o}C with gentle stirring for 2-4 h on hot plate. After stirring for 2-4h, reaction mixture allowed to cool to room temperature. Zno nanopowder is synthesed by sol gel process where Zinc acetate [Zn(CH3CO2)2.2H2O] and NaOH are used as a precursor material and the solvent, respectively. Zinc acetate is dissolved in NaOH by the molar ratio of 1:85. After stirring the solution with reflux at 70^{0}-75^{o} C for 4 hours.

Then filtration is done by whatman filter paper. During the filtration solution is washed by ethanol many times to avoid the impurities. After filtration, filtered sample is heated at 90^{o} C in oven for 2 hours. Heated sample is grinded and characterized under many techniques (like EDX, FTIR, SEM, etc.).

IV. DOPING OF DIDYMIUM

ZnO was doped with rare earth material didymium (a mixture of neodymium and praseodymium). Didymium is a naturally occurring element with major constituent Nd and Pr. The tables below shows various samples of Zno doped with didymium in different ratios. Consider the other elements varying ratios with didymium doping to ZnO

TABLE I
ZNO WITH 0.5% OF DIDYMIUM

Chemical name	Chemical formulae	Mole required	Material taken
Zinc acetate	(C2H3O2) 2Zn.2H2O	0.02	4.14 gm
Sodium hydroxide	NaOH	0.08	3.20 gm
Ethanol	C2H5OH	----------	100 ml
Didymium	Di	0.5	0.25 gm

TABLE II
ZNO WITH 1% OF DIDYMIUM

Chemical name	Chemical formulae	Mole required	Material taken
Zinc acetate	(C2H3O2) 2Zn.2H2O	0.02	3.89 gm
Sodium hydroxide	NaOH	0.08	3.20 gm
Ethanol	C2H5OH	----------	100 ml
Didymium	Di	1	0.5 gm

TABLE III
ZNO WITH 3% OF DIDYMIUM

Chemical name	Chemical formulae	Mole required	Material taken
Zinc acetate	(C2H3O2) 2Zn.2H2O	0.02	2.89 gm
Sodium hydroxide	NaOH	0.08	3.20 gm
Ethanol	C2H5OH	----------	100 ml
Didymium	Di	3	1.5 gm

TABLE IV
ZNO WITH 5% OF DIDYMIUM

Chemical name	Chemical formulae	Mole required	Material taken
Zinc acetate	(C2H3O2) 2Zn.2H2O	0.02	1.89 gm
Sodium hydroxide	NaOH	0.08	3.20 gm
Ethanol	C2H5OH	----------	100 ml
Didymium	Di	5	2.5 gm

V. RESULTS AND DISCUSSION

In order to identify the crystalline structure of the Didymium doped ZnO the Chemical composition and morphology of the samples was carried out using a scanning electron microscope (SEM), model Hitachi S-3400N. The compositional analysis is done by energy dispersive X-ray spectra (EDX) by thermo electron corporation IR absorption measurements were done using Shimadzu FTIR spectrometer .

a. SEM images of Didymium of ZnO

SEM images of the Di doped ZnO are shown in the figure 2. SEM shows that the Di doped ZnO has well ordered morphology, has low aggregation and homogeneous distribution of particle size The definite morphology was observed at all concentration. It appeared that the small grains were forming a cluster as evident from the SEM images (a), (b), (c) and (d) shown in figure 2.

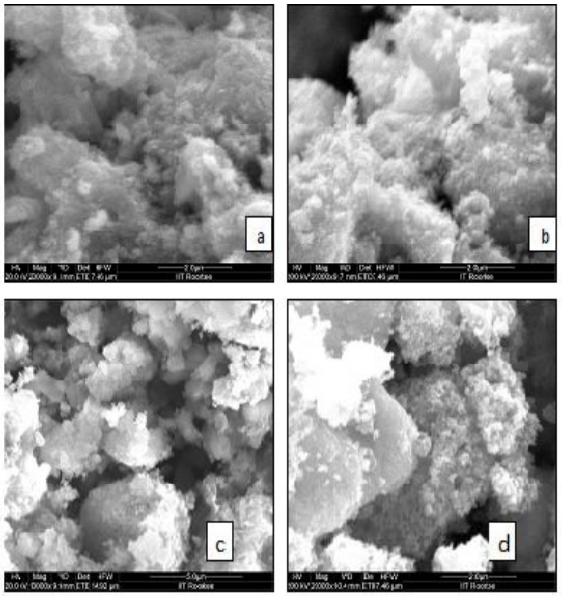

Fig. 2 SEM images of (a) 0.5%, (b)1%, (c) 3%, (d) 5% Di doped ZnO at 20,000X

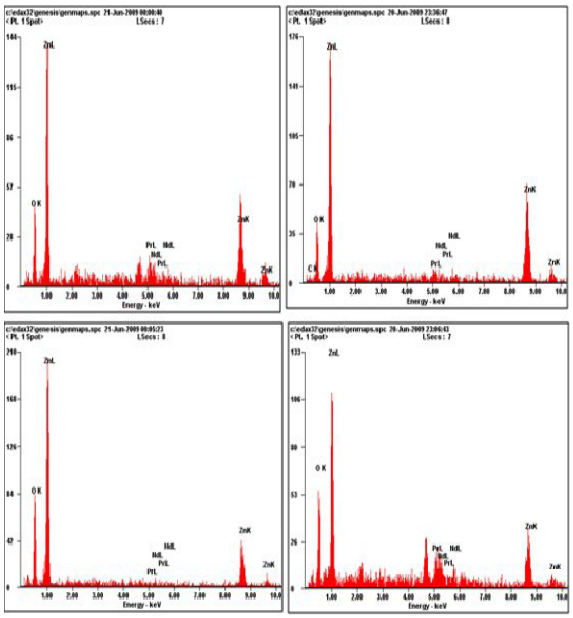

Fig. 3 EDX spectra of the Di doped ZnO (a) 0.5% Di, (b) 1% Di, (c) 3% Di, (d) 5% Di

The EDX spectra of the Di doped samples is shown in figure 3. The concentration of the different element observed, was listed in the table V. We observed that the all the samples are rich with Zinc and Oxygen element along with the small amount of Nd and Pr. The percentage of the Nd (Neodymium) and Pr (Praseodymium) increase at higher concentration due to the residue of unreacted part of Di at these concentrations.

TABLE V
LIST OF ELEMENTS PRESENT IN SAMPLES, DOPED WITH DIFFERENT CONCENTRATION OF DI (NOTE THAT FOR 0.5% DOPING THERE IS NO TRACES OF OTHER ELEMENTS)

Element	1 % Di		3 % Di		5 % Di	
	Wt%	At%	Wt%	At%	Wt%	At%
Ok	11.85	17.07	29.36	64.1	17.83	54.53
PrL	6.15	31.78	2.17	.54	19.58	6.80
NdL	3.95	1.87	3.71	.90	19.97	.677
Znk	73.27	48.09	64.76	34.6	42.62	31.90

b. FTIR spectra of Di doped ZnO Calcined at 4000C

FTIR spectra of Di doped sample at concentration 0.5%, 1%, 3%, 5% is shown in figure 4 respectively. No significant change are observed in these samples as compared to the pure synthesized ZnO.

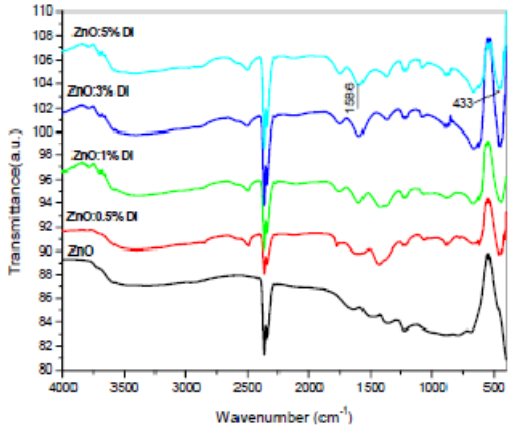

Fig 4.Comparison of didymium doped ZnO at different concentration after calcination at 400^{O}c

VI. CONCLUSION AND FUTURE SCOPE

We have successfully synthesized the single phase ZnO by using Sol gel method in basic medium having particle size about 4.8 nm. The synthesized system is also having band gap of 3.24 eV which is comparable with the standard value. We successfully dope the ZnO with Di ion up to 1% concentration .The XRD pattern shows the formation of pure phase of ZnO having wurtzite hexagonal structure. But at higher concentration of Di (3%, 5%) the system shows mixed phase as evident from the XRD analysis. SEM shows that the Di doped ZnO has well ordered morphology, has low aggregation and homogeneous distribution of particle size The definite morphology was observed at all concentration. There are possible applications of ZnO doped Dydmium in micro electromechanical systems (MEMS), both in sensors, actuators and in the fabrication of acoustic and electro-optical devices. In particular, it can be used as bulk acoustic wave (BAW) resonators and as thin film bulk acoustic wave (FBAR) resonators or surface acoustic wave (SAW) resonators. Moreover it is used in a variety of technical applications, including porcelain enamels, heat resisting glass, an activator in vulcanization, an additive for rubber and plastics, pigment in paints with UV-protective and fungistatic properties, spacecraft protective coatings, a constituent of cigarette filters, healing ointments, in optical waveguide, and many more.

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