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# Spectroscopic Study of Zinc Oxide additive to Tin Oxide Composites with Structural Evidences

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***Abstract:*** ***Thick films of pure $SnO_2$ and composite 1%, 3%, 5%, 7% and 9 % ZnO incorporated into pure $SnO_2$ were prepared by standard screen printing technique. All samples were dried and fired at $500^0$C at 5 hours. Thick films of $SnO_2$ and composite 1%,3%,5%,7% and 9 % ZnO incorporated into pure $SnO_2$ were characterized by SEM, EDAX, XRD, FTIR and UV to study surface morphology, elemental analysis, crystalline Phases of films, vibrational and spectroscopic study respectively. The UV-visible spectroscopic study was undertaken in terms of absorbance and absorption coefficient with percentage composition.***
***Keywords: SEM, absorbance, absorption coefficient, UV-visible spectroscopic study etc.***

## I. INTRODUCTION

Screen printing technique was introduced in the later part of 1950s. It is versatile technique to produce robust, compact and relatively inexpensive hybrid circuit for different applications [1, 2]. Different methods have been investigated to grow pure $SnO_2$ and composite 1%, 3%, 5%, 7% and 9% ZnO incorporated into pure $SnO_2$ such as Spray pyrolysis, Vaccum evaporation, chemical vapour deposition, magentron sputtering, pulsed laser deposition, sol-gel technique and screen printing technique[3, 4].
Zinc oxide is transparent conducting oxide (T.C.O.), which has been studied extensively. It has wide band gap of 3.3 eV. It is demanded material for the application in gas sensor, electronic displays, manufacture of blue light emitting diodes and surface acoustic waves [5]. Tin oxide ($SnO_2$) and doped tin oxide ($SnO_2$) is also been studied in different forms like powder form to bulk ceramics. Pure tin oxide ($SnO_2$) has wide band gap 3.6 eV with n-type semiconductor [6]. Additionally $SnO_2$ and ZnO material is chemically stable, easy to fabricate, non-toxic and low cost [7]. In 2018 Mursal et al., has shown that with increase in temperature from $300^0$C to $700^0$C there is band gap decrement from 3.82 eV to 3.69 eV is observed for ZnO thin films deposited by sol-gel spin coating method, in this case optical absorption coefficient was relatively high (up to $5\times10^4$ $cm^{-1}$)[8]. In our paper annealing temperature and annealing time is constant for all pure and composite films of ZnO:$SnO_2$ but absorption coefficient value changes with composition of composite material which is due to morphology of annealed films and particle size effect on the absorbance.

## II. MATERIALS AND METHODS

In this paper standard screen printing method has been adopted to prepare thick films of pure $SnO_2$ and composite 1%,3%,5%,7% and 9% ZnO incorporated into pure $SnO_2$. AR grade (99.9% purity) pure $SnO_2$ and ZnO was used. Screen having quadrilateral shape of 100 maize size and glass substrate having dimension 2cm×1cm×1mm as support used for adhere film material at high temperature had been employed. Ethyl cellulose, glass frit and butyl carbittol acetate was properly mixed to form paste in such a way that so as to control morphological, thixotropic and rheological properties of paste. The prepared paste was then screen printed through mask position by squeegee on the glass substrate [9]. The prepared thick films were dried under IR lamp for 3-4 hours subsequently followed to annealing in muffle furnace for 5h at $500^0$C. The thick films were characterized for X-ray diffraction, Scanning electron microscopy (SEM), Fourier transform Infra-red spectroscopy and UV-Visible spectrophotometry.

## III. RESULT AND DISCUSSION

### *A. Structural Characteristics*

*1) X-ray Diffraction Analysis:* In order to understand the crystalline phases in $SnO_2$ and their additive zinc oxide thick film samples prepared by standard screen printing method, XRD analysis was performed using CuKα radiation of wavelength λ=1.54018 Å at accelerating voltage of 40 kV and 40 mA current in continuous scan mode with 15 degree/min at room temperature. Fig. 1 shows an XRD pattern of $SnO_2$ and composite $SnO_2$: ZnO thick film samples in 1%, 3%, 5%, 7%, and 9% plotted in the range 20-80 position (2θ) on X-axis verses intensity on Y-axis. All Peaks of different composition are observed at the different angular positions which were indexed to the (110), (101), (200), (111), (211), (220), (310), (112) and (301) respectively. The observed peaks match well with the reported data of Tin-oxide (JCPDS Card No. 41-1445) confirming the polycrystalline nature.

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The higher peak intensities in XRD pattern at peak position 110 is due to the better crystalinity and bigger grain size can be attributed to the agglomeration of particles. The narrow and sharp peaks reveal that $SnO_2$ and $ZnO:SnO_2$ compositions are well crystallized. It has been observed that peak intensity value either decreases or increases with effect of additive material incorporated into tetragonal phase of tin oxide. The ZnO (100) is present near the $SnO_2$ (200) peak confirming the segregation of ZnO was evident from the XRD pattern diffraction peaks near low intensity [7, 10] with wurtzite hexagonal phase (JCPDS Card No.36-1451). In 3% XRD spectrum ZnO (102) peak is more prominent near tetragonal $SnO_2$ (211) [11]. The intensity decrement and peak shifting higher or lower 2θ angle indicates the change in crystal parameters, crystallite sizes, d-spacing and unit cell volume. The reason behind less or more intensity along each plane is explained in terms of different atomic or molar density present along every plane of composite material. Average crystallite size was calculated using Scherer equation [7, 10-11] (equation 1) was found in between 13 nm to 39 nm.

$$D = \frac{0.9\lambda}{\beta COS\theta} \qquad \text{..... (1)}$$

Where, D- is the average grain size,
λ=1.542 Å (X-ray wavelength),
β=the peak FWHM in radiation and
θ=diffraction peak position in terms of angle.

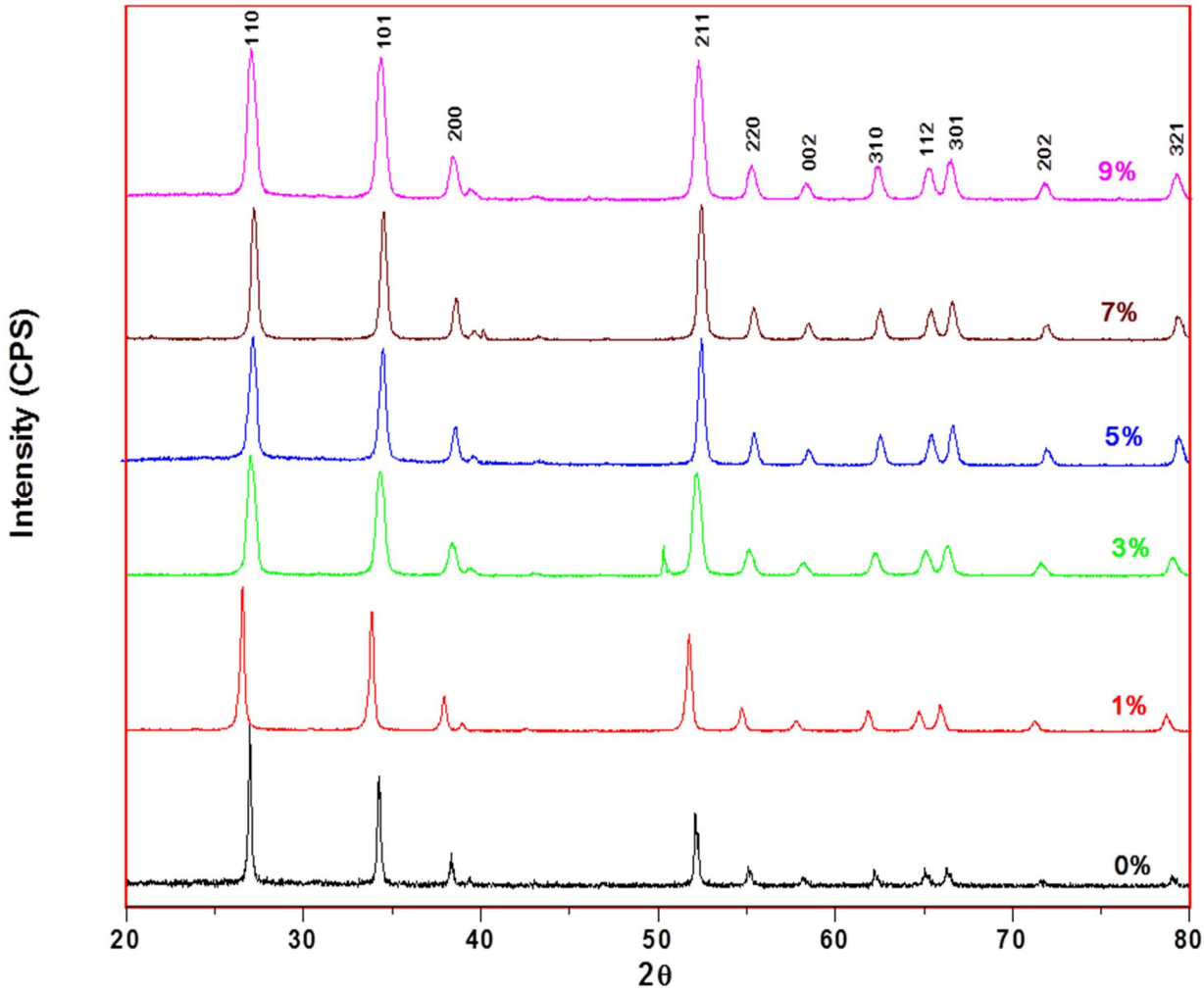


Fig 1: XRD spectra of (a) black line-Pure $SnO_2$ (b) red line-1% $ZnO:SnO_2$ (c) green line-3% $ZnO:SnO_2$ (d) blue line-5% $ZnO:SnO_2$ (e) wine red-7% $ZnO:SnO_2$ (f) pink line-9% $ZnO:SnO_2$

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2) *Morphological evedences with Scanning Electron Microscopy:* Scanning electron microscopy imparts high-resolution imaging useful for evaluating various materials to surface fractures, flaws, comtaminants or corrosion. Focussed beam of secondary electrons interact with atoms in the sample produces various signals that consists of information about the surface topography and composition of sample[7]. All images were scanned at 10000x with 5 µm dimension scale using Nova NanoSEM NPEP 303. SEM was useful to collect the information regarding the morphological properties of deposited screen printed films of pure $SnO_2$ and composite 1%, 3%, 5%, 7% and 9 % ZnO incorporated into pure $SnO_2$. The following SEM images reveals that all film images are porous in nature with interconnected pores. The avarage grain size of scanned images in between 45 nm to 74 nm. Among all composition 5% ZnO:$SnO_2$ have least size (47.65 nm). The Grain size of nanomaterial is inversly proportional to avarage surface area. The exposed grain size for any physical or chemical property measurement is a function its surface area. The least value of grain size shows maximum available surface area for absorbance of pure and composite ZnO:$SnO_2$.

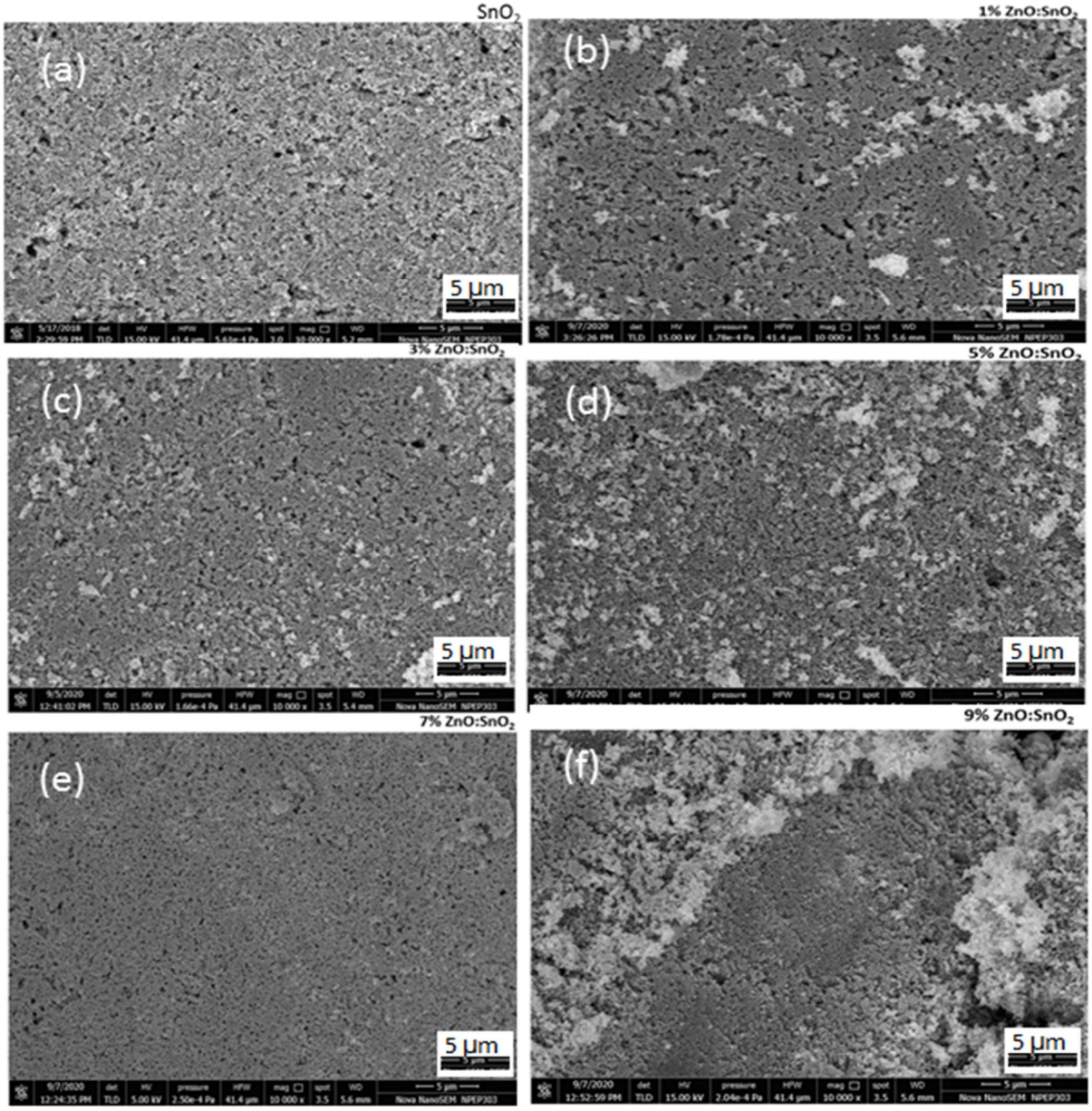


Fig 2: SEM micrographs corresponding to (a) Pure $SnO_2$ (b) 1% ZnO:$SnO_2$
(c) 3% ZnO:$SnO_2$ (d) 5% ZnO:$SnO_2$ (e) 7% ZnO:$SnO_2$ (f) 9% ZnO:$SnO_2$

The morphological and particle size has dominant effect on the screen printed zinc oxide composites of tin oxide. The recent study of influence of size and morphology on optical properties of carbon nanostructures have investigated by Hajar Sadeghi et al [12].

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3) *Ftir Spectroscopy:* FTIR is useful technique as qualitative and quantitative tool for organic and inorganic samples. It is useful for solids, liquids and gaseous samples and measures the wide range of wavelengths in IR spectra that are absorbed by the material. The Ftir is done by following standard method of solid phase KBr pellet technique. The FTIR spectra is recorded at room temperature for pure $SnO_2$ and composite 1%, 3%, 5%, 7% and 9 % ZnO incorporated in pure $SnO_2$ in IRAffinity-1 Shimadzu FTIR Instrument. A small amount 2 to 5 mg sample powder is mixed in 300 mg Potassium Bromide (KBr) of spectroscopic grade purity made up of UVASOL Company to form clear transparent 13mm circular pallet or disc with 1mm thickness when mixture was pressed in KBr Press Model M-15 at a pressure of about 5×106 Pa in an evacuated die. The following Ftir spectra were recorded between 400 $cm^{-1}$ to 4000 $cm^{-1}$ consist of transmittance at different wavenumber.

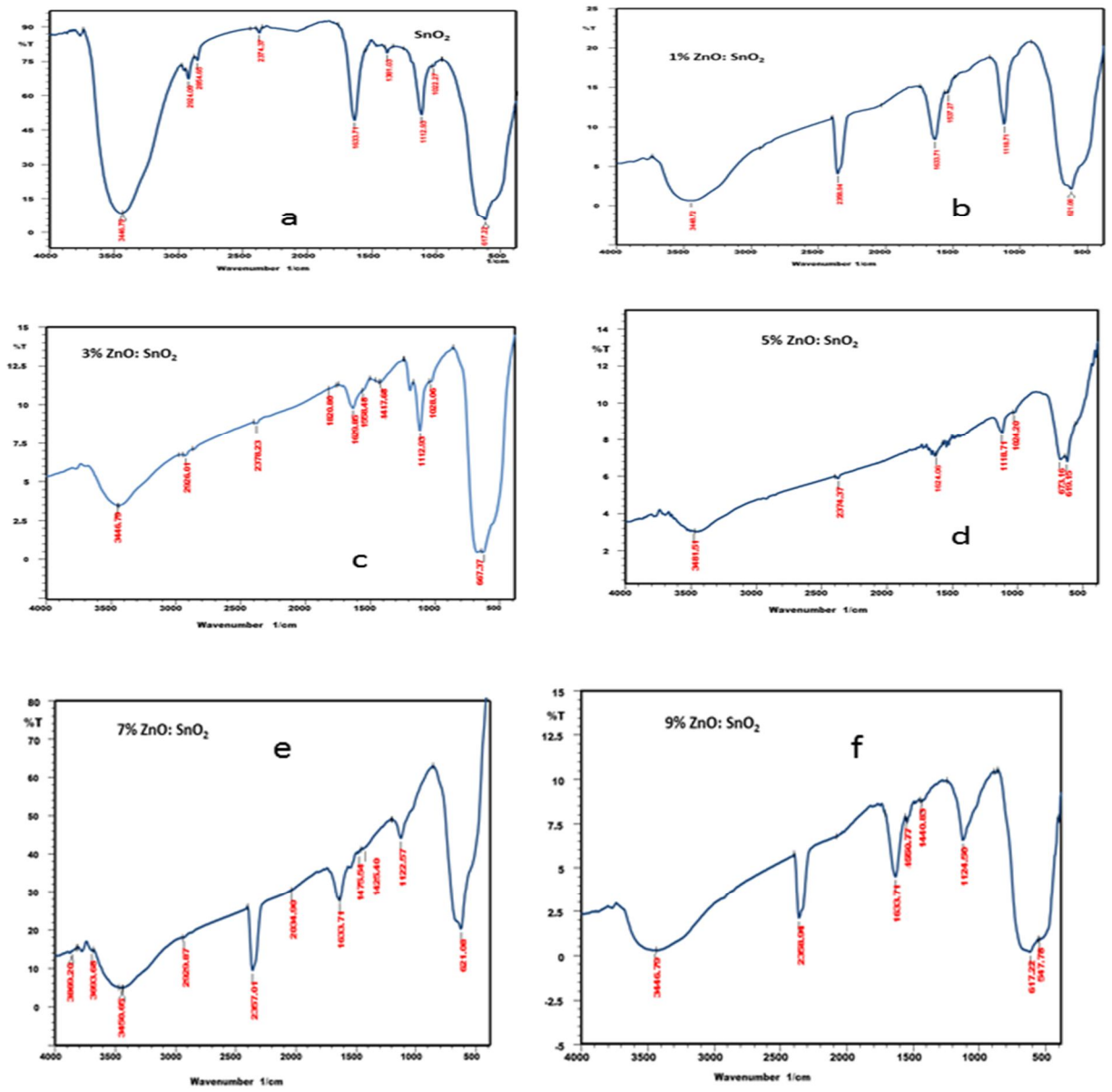


Fig 3: FTIR spectra of corresponding to (a) Pure $SnO_2$ (b) 1% ZnO: $SnO_2$ (c) 3% ZnO:S $nO_2$ (d) 5% ZnO:$SnO_2$ (e) 7% ZnO:$SnO_2$ (f) 9% ZnO:$SnO_2$

From fig. 3 it has been evident that the absorbance band at the extreme left at 617cm-1 to 667cm-1 and approximately 480cm-1 to 510 cm-1 corresponds to Sn-O and Zn-O bonds confirming the ZnO and $SnO_2$ nanoparticles. The IR bands at the 3446cm-1 to 3480 cm-1 corresponds to O-H confirming the presence of hydroxyl groups on the surface of thick films of pure $SnO_2$ and composite 1%, 3%, 5%, 7% and 9 % ZnO incorporated into pure $SnO_2$[13].

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4) *UV Spectroscopic Study:* UV-Visible Absorption spectroscopy is intense non-destructive testing technique for exploring the properties of semiconductor nanomaterial. Absorbance may depend on nature of surface, oxygen deficiency, band gap and impurity centers. The UV absorbance of pure $SnO_2$ and composite 1%, 3%, 5%, 7% and 9 % ZnO incorporated into pure $SnO_2$ was recorded on UV spectrophotometer 2012 made analytical technology Ltd. 1mg of all sample powder is dissolved into 10ml 1N $H_2SO_4$ as a solvant to form clear solution followed by 10 minutes sonication for all samples. A cuvette of 1 $cm^3$ was employed for measurment with scan step 5 nm with wide range of 200 nm to 800 nm. Duterium amd Tungston lamp was used as a sorce of radiation for broad range with photo-dioade array detector. A solvant was emplyed for dissolution purpose. The solvant effect was first nullified as blank reading. Zero slovant absorbnace line clearly indicates that there is no effect of solvant on the absorbance spectra of pure $SnO_2$ and composite 1%,3%,5%,7% and 9 % ZnO incorporated into pure $SnO_2$. Particle size and morphology has pronounced effect on the spectoscopic properties on the basis of physical properties like absorption[7-8]. The absorption coefficient α is related to absorbance and thickness of material by following equation[12, 14].

$$\alpha = \frac{2.303 * A}{d} \quad \text{.....(2)}$$

Where, A=Absorbance of material,
d= Path length of cuvette or cell

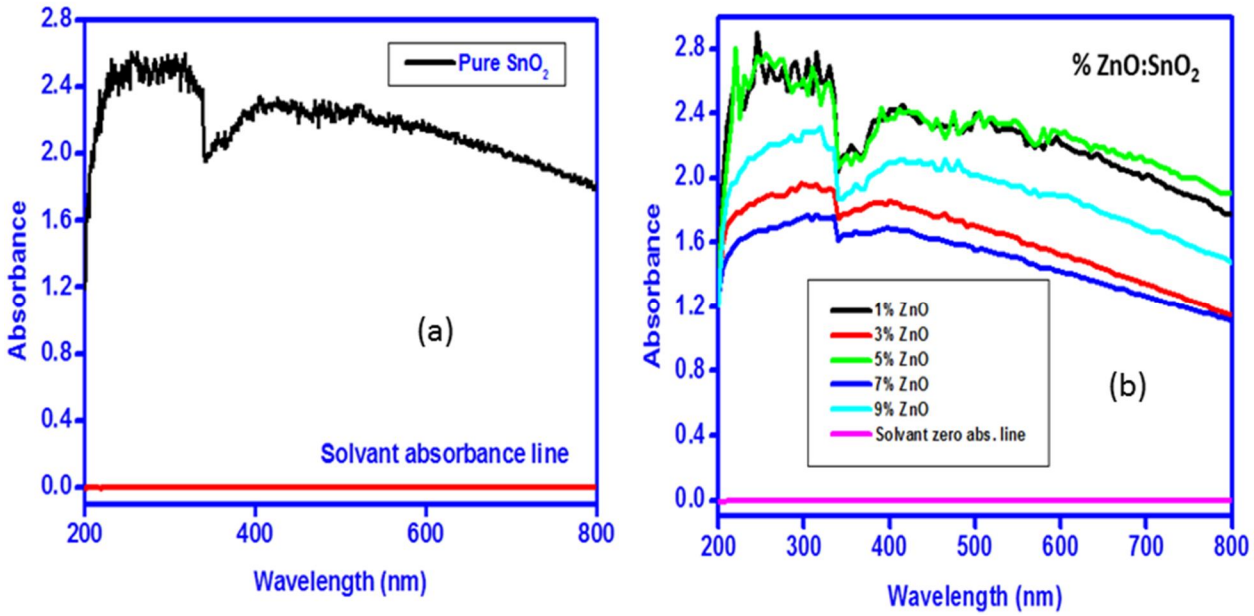


Fig 4: UV-Visible spectra of (a) Pure $SnO_2$ (b) composite % ZnO: $SnO_2$ and zero absorbance line of solvent

From the absorbance spectra of pure SnO2 (fig a) and composite ZnO:$SnO_2$ (fig b) there are intense line graph is observed consisting intense zigzag and shoulders peaks. At the bottom, solvent line is observed suggesting blank-zero absorbance line. The variations in absorbance are due to better degree of crystallization from composition to composition with pure $SnO_2$ as indicated in XRD and SEM. Following table gives the compositional variation for Absorbance and absorption coefficient (α).

| composition | Wavelength (nm) | Absorbance (A) | Absorption coefficient (α) $cm^{-1}$ |
|---|---|---|---|
| 0 Pure $SnO_2$ | 255 | 2.6005 | 5.988952 |
| 1% ZnO | 315 | 2.7805 | 6.403492 |
| 3% ZnO | 300 | 1.9656 | 4.526777 |
| 5% ZnO | 200 | 2.8042 | 6.458073 |
| 7% ZnO | 315 | 1.7775 | 4.093583 |
| 9 % ZnO | 320 | 2.3159 | 5.333518 |

Table.1. Compositional variation for Absorbance and absorption coefficient (α)

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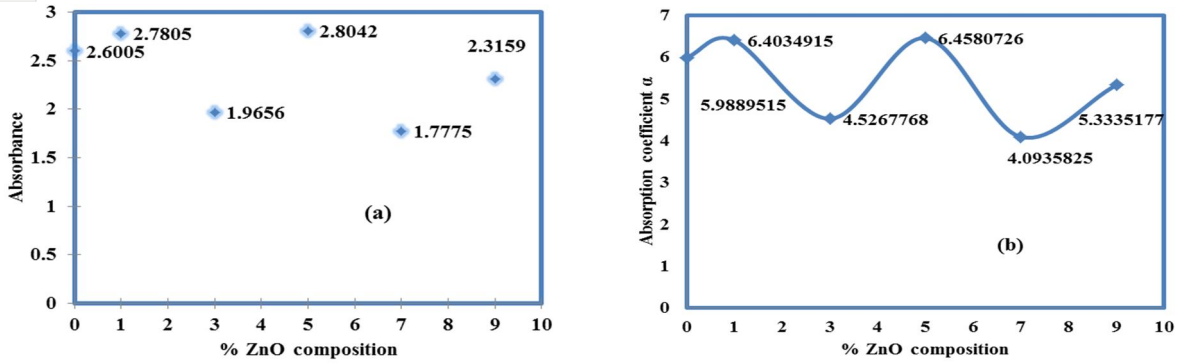


Fig 5: Spectroscopic graph of (a) absorbance Vs. % Doping Concentration of composite % ZnO:$SnO_2$ (b) Absorption Coefficient Vs. %Doping Concentration of composite % ZnO:$SnO_2$

The graph of absorbance and absorption coefficient as a function of % doping or zinc oxide additive concentration is plotted, there are variations among absorbance and absorption coefficient values are observed. The maximum values for absorbance and absorption coefficient are 2.8042 and 6.45 $cm^{-1}$ for 5% ZnO:$SnO_2$ composition. The value of absorption coefficient was approximately matched with result obtained for carbon nanostrucures in different liquids[12]. The absorption coefficient is least for 7% ZnO:$SnO_2$ and maximum for 5% ZnO:$SnO_2$.

## IV. CONCLUSION

In this research, thick films of pure $SnO_2$ and composite 1%,3%,5%,7% and 9 % ZnO incorporated into pure $SnO_2$ has succesfully deposited using standard screen printing method. We found that Particle size and morphology has pronounced effect on the spectoscopic properties. Ftir spectra reveals the presence of composite ZnO/$SnO_2$ material. High absorption coefficient values are responsible to absorb more photons. 5% ZnO-$SnO_2$ shows high absorbance and absorption coefficient indicates that better amount of crystallinity is present in 5% ZnO:$SnO_2$ sample.

## V. ACKNOWLEDGEMENTS

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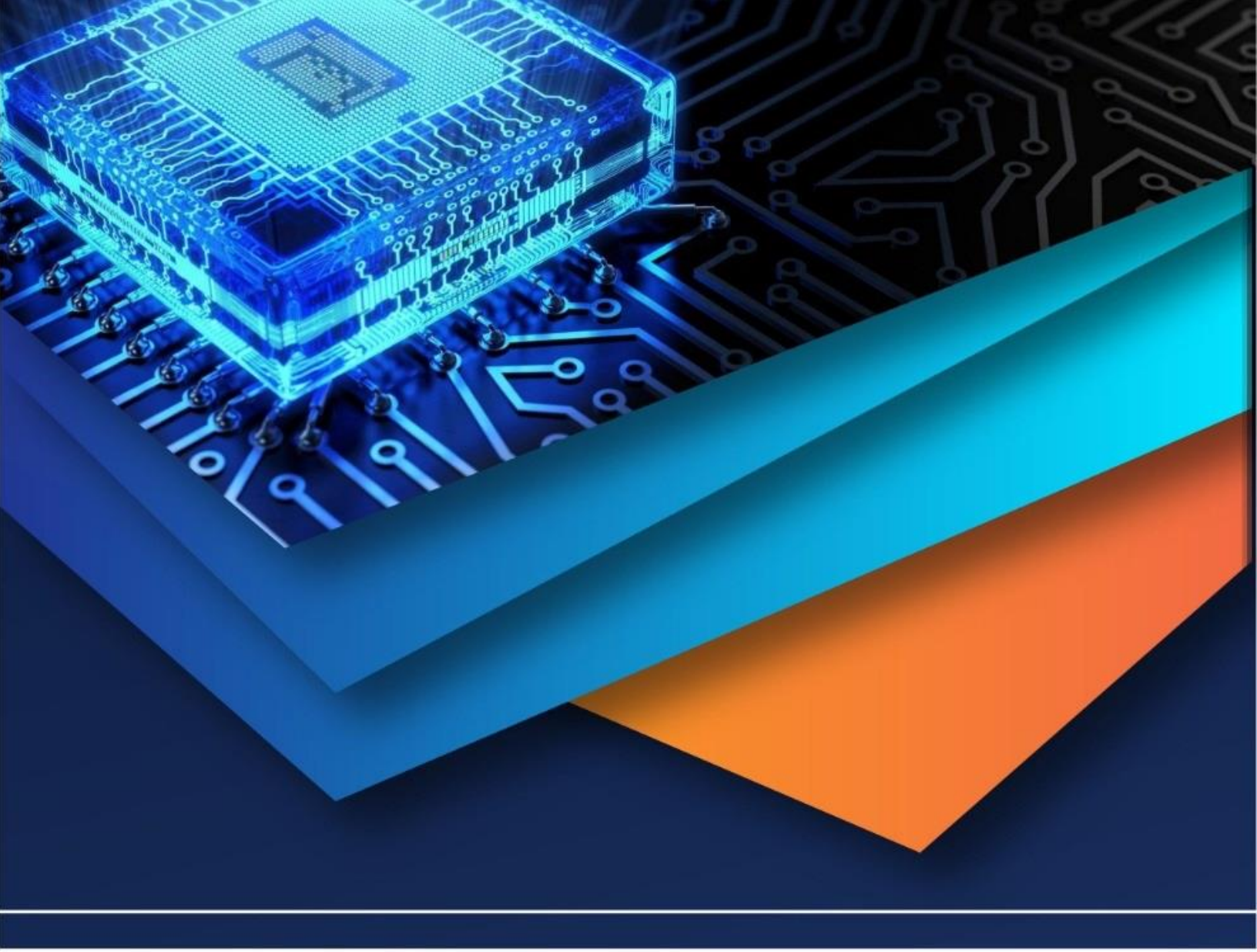

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