SPECTROPHOTOMETRIC STUDY OF SCREEN PRINTED %CuO:SnO2 NANO-COMPOSITE POWDER FIRED AT HIGH TEMPERATURE

R.M. Nikam^{*1}, K.H. Kapadnis² and R.Y. Borse³

*1Department of Chemistry, M. J. M. ACS College, Karanjali, Tal- Peth, Dist-Nashik, MS, India ²Department of Chemistry, L. V. H. ACS College, Panchavati, Dist-Nashik, MS, India ³Department of Physics, M. J. M. ACS College, Karanjali, Tal- Peth, Dist-Nashik, MS, India *1rohitnikam9@rediffmail.com

ABSTRACT

Thick films of pure SnO_2 and composite 1%, 3%, 5%, 7% and 9% CuO incorporated into pure SnO_2 were prepared by standard screen printing technique. All samples prepared on glass support/substrate were dried and fired at $500^{\circ}C$ at 5 hours in muffle furnace. Thick films of SnO_2 and composite 1%, 3%, 5%, 7% and 9% CuO 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 spectrophotometric parameters such as absorbance and absorption coefficient were studied. The UV-visible spectrophotometric study was undertaken in terms of absorbance and absorption coefficient withvarying percentage composition.

Keywords: morphology, spectrophotometric study, absorbance, absorption coefficient, etc.

Introduction

It has been evident that screen printing technique which was introduced in the later part of 1950s is most popular and versatile chemical method to deposit thick films on glass or alumina substrate described by Krishnan et al.,(2005). According to Guidi et al., (2002) screen printing technique is popular technique to construct robust, compact and relatively cheap hybrid circuit for different applications like gas sensor, electric appliances, transistors and optical devices. Different methods have been investigated and developed to grow pure SnO₂and differentcomposites material ofCuOincorporated into pure SnO₂ such as pyrolysis, Vaccum evaporation, Spray chemical vapour deposition, magentron sputtering, pulsed laser deposition, sol-gel technique and screen printing technique as stated in Joseph et al., (1996) & Jaydev et al., (1998). Among all screen printing method has found to gain popularity for the formation of superconducting oxide films. Borse R. (2008) in his reference book talked about the advantages of thick film techniques includes fast processing, economical use of paste and low cost. Being metal oxides have nonstoichiometric structure, defects in crystal lattice, different morphology and crystallite pattern finds a great deal in electrical and optical devices. The result demonstrated by Shukla (2012), on absorption spectra of tin

oxide nanoparticles obtained in UV-Visible region shows blue shift in the absorption edge at 268 nm as compared to bulk. The relative peak shift is useful in different applications like thick film resistor and gas sensing. As discussed by Verma et al., (2013) and Hassan et al., (2019) The band gap and absorption edge modification of tin oxide takes place when different dopant material like CuO, ZnO, TiO₂, WO₃, ZrO₂ and many other binary and ternary phase are added into different varying concentration. The good results are obtained by Dhineshbabu et al., (2016) in their research and put forward that pure CuO has article absorption edge at 219 nm when CuO nanoparticles were prepared from sonochemical process; this is because of direct transition of electrons. In this paper a nano CuO:SnO₂composite powder sample of material has investigated for structural and spectroscopic analysis in terms of absorbance and absorbance coefficient with minimal concentration to maximum concentration in solvent phase similarly described into Hazar S. et al (2016) about solvent phases.

Materials and methods i. Preparation of SnO₂ Thick Film

Tin-oxide and composite thick films of cuprous oxide (CuO)blended in tin oxide with percentage composition were prepared on glass substrate with particular dimension by using standard screen-printing technique (Ansari et al., 1996, Prudenziati et al., 1996, Harper. 1974, Kiran et al., 2006, Nimal et al., 2004. and Patil et al., 1998). The analytical grade SnO₂and CuOchemical powder (99.99 %) was weighed. The calcined SnO₂and CuO powder of different composition was mixed and crushed thoroughly with glass frit which acts as permanent binder and ethyl cellulose acts as a temporary binder. The mixture was then mixed with butyl carbittol acetate as organic vehicle to form the paste. The paste was then screen printed onto the surface of glass substrate. The detail of the technique was described in reference book of Harper. (1974). After screen printing the films were dried under ordinary visible lamp of 200 watt for 2-3 hr. followed with firing into muffle furnace, dried under air at 500° C for 5 hr. After atmosphere preparation ofpure SnO₂ and CuO-SnO₂ composite thick films were subjected to structural, morphological and spectroscopic study.

ii. Structural, Morphological and spectroscopic characterization

a. Using X-ray diffraction (Miniflex Model, Rigaku, Japan) analysis from 20-80, 20 was carried out to examine the crystalline phases of the SnO₂ films samples. The instrumental broadening was removed using silicon standard sample. The single line approximation method has adopted for the crystallite size determination using high intense peak. The average grain size of pure and composite tin oxide thick films sample werecalculated by using the Scherer formula as referred on behalf of Chatterjee et al., (2003):

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

Where, D-average grain size, λ -1.542 Å (X-ray wavelength), β - Peak FWHM θ -Peak position.

b. The microstructure and chemical composition of the films were analyzed using a scanning electron microscope [Nova nano SEM NPEP303] coupled with an energy dispersive spectrometer (EDS JEOL, JED-2300, Germany).

- c. 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 SnO₂ and composite 1%,3%,5%,7% % and 9 CuO mixed in pure Shimadzu SnO₂inIRAffinity-1 FTIR Instrument. A small amount 2 to 5 mg sample powder is mixed in approximate 250mg-300 mg Potassium Bromide (KBr) of spectroscopic grade purity made up of UVASOL Company to form clear transparent proper 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 spectral resolution of 2 cm⁻¹ and taking 32 scans for each sample.
- **d.** UV 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 % CuO incorporated into pure SnO₂ was recorded on UV spectrophotometer 2012 made analytical technology Ltd. 1mg of all sample powder is dissolved into 10ml 1N H₂SO₄ as a solvant to form clear solution followed by 10 minutes sonication for all samples. A cuvette of 1 cm³ 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 solvent effect was nullified as blank reading. The absorption coefficient α is related to absorbance and thickness of material by following equation as reffered on behalf of Hajar et al., (2016) and ahmed et al., (2011).

 $\alpha = \frac{2.303 * A}{d}(2)$ Where, α -absorption coefficient A-Absorbance of material, d- Path length of cuvette or cell

Result and discussion

a. XRD

X-ray diffraction is the plot of peak position with intensity along the spectra. The screen printed thick film of additives of CuO and tin oxide (SnO₂) base material was highly crystalline and all are well oriented along planes (110), (101) and (211). It confirms the presence of polycrystalline sample mixtures. The presence of CuO (002) was evident from the mixture in terms of asterisk near (101) plane of tin oxide which was confirmed by Verma et al., (2013). It has been depicted from the figure the polycrystalline nature of spectrum well orient in (110) plane. According to Hassan et al., (2019) all observed peaks are well agreement with JCPDS card no. 41-4145. The higher peak intensities in an XRD pattern along the spectrum are due to better crystalinity grain bigger size attributed and to agglomeration of nanoparticles as depicted in SEM images also. The molar density along the spectra is less or more hence observed peaks have low or denser in their intensities. The crystallite sizes were calculated using Scherer formula (eq.1) lies in the range of 11nm to 20nm.



Fig 1: XRD spectra of (a) Pure SnO_2 (b) 1% CuO: SnO_2 (c) 3% CuO: SnO_2 (d) 5% CuO: SnO_2 (e) 7% CuO: SnO_2 (f) 9% CuO: SnO_2

b. Scanning Electron Microscopy and EDAX analysis

Scanning electron microscopy imparts highresolution imaging utilized 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 as described by Karthik et al., (2008).All images were scanned at 10000x with 5 μ m dimension scale. The following SEM images reveals that all nanocomposite images are well agglomerated meso-porous in nature with interconnected pores. Some of the images have miniature crack into their surface nature. The avarage grain size of scanned images in between 53 nm to 67 nm. The average grain size of nanomaterial is inversly proportional to its avarage surface area. The exposed grain size for any physical or chemical property like absorbance measurement is a function of its surface area.



Fig 2: SEM micrographs corresponding to (a) Pure SnO_2 (b) 1% CuO:SnO₂ (c) 3% CuO:SnO₂ (d) 5% CuO:SnO₂ (e) 7% CuO:SnO₂ (f) 9% CuO:SnO₂

Elemental analysis by EDAX is useful to explain quantitative measurment of elemental analysis and present of qualitative parameter to study the presence of nanomaterial. From fig. 3 it has been concluded to presence of pure SnO₂ and composite CuO-SnO₂nanomaterial in appropriate mixture, indicating percentage composition were succesfully prepared.



Fig 3: EDAX spectra of corresponding to (a) Pure SnO_2 (b) 1% CuO:SnO₂ (c) 3% CuO:SnO₂ (d) 5% CuO:SnO₂ (e) 7% CuO:SnO₂ (f) 9% CuO:SnO₂

c. Ftir Analysis

Functional property of composite CuO-SnO₂is investigated in fig. 4. The presences of characteristic frequencies are shown in following figure. It has been evident that the absorbance band at the extreme left at 617cm⁻ ¹to 671 cm⁻¹ and 381 cm⁻¹to 410 cm⁻ ¹corresponds to Sn-O and Cu-O bonds confirming SnO₂ and CuO nanoparticles. The are concurrent as reported results bv

Dhineshbabu et al., (2016), Lande et al., (2015) and Sagadevan et al., (2016). The IR bands at the 3450 cm⁻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 CuO incorporated into pure SnO_2 . The IR band at 1629-1637 cm⁻¹attributes to deformation mode of OH groups described by Sagadevan et al., (2016).



Fig 4: FTIR spectra of corresponding to (a) Pure SnO_2 (b) 1% CuO:SnO₂ (c) 3% CuO:SnO₂ (d) 5% CuO:SnO₂ (e) 7% CuO:SnO₂ (f) 9% CuO:SnO₂

d. Spectroscopic analysis

Spectrophotometer can be divided into according to wavelength and application context. The absorbance of CuO-SnO₂nano

powder with different composition has depicted in following figure



Fig 5: UV-Visible spectra of (a) Pure SnO₂ (b) composite % CuO: SnO₂ and zero absorbance line of solvent

It has been clear to view the zigzag nature of graph. The solvant effect was first nullified as blank reading. path length of cylindrical cuvette/cell was 1 cm³. 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% CuO incorporated into pure tin oxide(SnO_2). The variation of absorption

coefficient with percentage compososition follows the linear graph which was inclined in downward direction as depicted in fig . 6. According to Karthik et al., (2008). and Mursal et al., (2018) particle size and morphology plays an important role on the spectoscopic investigation of different constituent of tin oxide composite material.

composition	Wavelength (nm) λ_{max}	Absorbance (A)	Absorption coefficient (α) cm ⁻¹
Pure SnO ₂	255	2.6005	5.9889515
1% CuO	290	2.2434	5.1665502
3% CuO	225	2.1637	4.9830011
5% CuO	205	1.7184	3.9574752
7% CuO	215	2.3136	5.3282208
9 % CuO	210	2.1282	4.9012446



Table.1.Percentage composition of Absorbance (A) and absorption coefficient (α).

Fig 6: Spectroscopic graph of (a) absorbance Vs. % Doping Concentration of composite % CuO:SnO₂ (b) Absorption Coefficient Vs. %Doping Concentration of composite % CuO:SnO₂

Conclusion

The screen printed thick films of cuprous oxide-tin oxide (CuO-SnO₂) nanomaterial fired at 500^{0} C for 5 hours are highly crystalline and porous as described in XRD and SEM study. Elemental analysis indicates the presence of cuprous oxide-tin oxide (CuO-SnO₂) powder. FTIR study reveals bonding and frequencies in

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