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# **Nigerian Journal of Materials Science and Engineering (NJMSE)**

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## Editorial Comment

It is a great pressure for the editorial board of the Nigerian Journal of Materials Science and Engineering (NJMSE) to present Volume 10 Number 1 of the journal for 2020 for the world research and development community.

The Materials Science and Technology Society of Nigeria (MSN), as a professional learned body, has made the publication of this research journal to be of very good quality and high standard comparable to any in her class. Our major thrust is to disseminate materials science and engineering and allied research activities from Nigeria, Africa and the world over. We are slowly and gradually impacting on the research community work with this specialised journal from a reputable learned and professional body in Nigeria. We are presently not insisting on number but we very much believe, with the thoroughness of our approach to the review and assessment process, we are convinced that with our resolve to publish quarterly, the board is convinced that more researcher would take advantage of this.

As a journal whose policy is to maintain the standard best practices and in addition to help young researchers to advance in the art and science of scientific findings dissemination, had faced tremendous challenges which were expected. It is heart-warming that we can look back and be glad to see the society publishing the 10<sup>th</sup> volume. These volumes and the previous ones would be available for FREE downloading on our society website ([www.msn.ng](http://www.msn.ng)) through a link prior to the specialised journal website to be available soon. Arrangements are in advanced stages for the hosting of this journal by reputable international online submission system are being worked on.

Volume 10 (2020) Number 1 consists of eight (8) high standard articles covering different specialised areas of materials research. It is our hope that this humble effort, presently by voluntary efforts of senior members of the Society, at disseminating research findings as put together in this volume which have contributed to the body of knowledge, would have enriched the information base and complemented Materials Research efforts from around the world.

We appreciate all our reviewers and associate editors involved for their prompt action on the manuscripts and cooperation as we look forward to submission of manuscripts which can be forwarded as detailed below.

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**AIM:** To improve the international exchange of scientific research in materials science and engineering.

## INTRODUCTION

The Nigerian Journal of Materials Science and Engineering (NJMSE) publishes reviews, full-length papers, and short communications recording original research results on, or techniques for studying the relationship between structure, properties, and uses of materials. The subjects are seen from international and interdisciplinary perspectives covering areas including metals, ceramics, glasses, polymers, composite materials, fibers, electronic materials, alternative energy materials, nanostructured materials, nanocomposites, biological, biomedical materials, etc. The NJMSE is now firmly established as the leading source of primary communication for scientists investigating the structure and properties of all engineering materials in Nigeria, Africa and the Rest of the World.

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NJMSE is introduced to publish research findings on current topical issues of interest to both public and private sectors. The scope of the Journal focuses on experimental, empirical and theoretical research in Materials Science and Engineering. Findings from multidisciplinary research covering diverse areas of interest with potential impact on the public and private sectors of both the national and international communities will be priorities of the journal. Our major focus is the use of Materials Science and Engineering principles to solve basic problems peculiar to African and the developing world while contributing to knowledge on the global scale.

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# Synthesis and Surface Characterisation of Cu-Doped Tin Oxide Thin Film for Optoelectronic Applications

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## Abstract

Copper doped tin oxide thin film was synthesised by electrodeposition technique. Film growth was maintained at cathodic potential of 1.60 V at a varied deposition time. Surface morphological studies of the deposited films were achieved by field emission scanning electron microscopy (FE-SEM). The scanning electron microscopy image revealed evenly distributed films, across the substrate with rice-like or dome-like particles, depending on the deposition time. Post-annealing enhanced films crystallinity and particles agglomeration. Energy disperse X-ray spectra revealed the elemental constituents present in the film. The results obtained from electrical characterisation of the samples showed the ohmic properties of the deposited sample. X-ray diffraction results indicated that samples are polycrystalline in nature with tetragonal rutile structure. The average interplanar spacing and crystallite size of the samples were estimated as 2.93 Å and 202.5 Å respectively. Optical characterisation of the samples showed that absorption and transmittance across the ultraviolet-visible spectrum range depend on deposition time. The estimated energy band gaps of 3.06 eV suggested the films as good candidates for transparent contact electrodes in optoelectronic applications.

**Keywords:** Electrodeposition, Annealing, Doping, Crystallinity, Morphology, Energy band gap

## INTRODUCTION

It has been noted that nothing compares with the sun energy potential. Solar power is readily abundant and efficient to supply the world's energy needs. Since solar energy can be sustained and renewed, its availability cannot be exhausted (Görig and Breyer 2016). Global warming is characterised by disastrous effects, consequently threatening its detrimental influence on climate, environment (with plants and animals) and human health (Resch *et al.* 2008). Significant source of greenhouse gases (GHG) are power plants (coal-fired).

These are accountable for about 25% of all anthropogenic emissions (Kabir *et al.* 2018), however, the emission of these gases during production of solar power is insignificant. The ratio of CO<sub>2</sub> emitted per KWh obtained from solar power, natural gas and coal are estimated to be 1:9.5:18 respectively (Machol and Rizk, 2013). This corroborates how friendly solar energy is to the environment when compared to other sources of energy (Weisser 2007). Solar power has a realistic solution to lessen global warming crisis, it can effectively replace coal and gas-based energy sources creating an environment with social and economic benefits for attaining sustainable development. Solar energy is a consistent, clean and pollution free source of energy. Distinct from other energy sources, its consumption is not accompanied by the release of injurious gases (e.g. volatile organic compounds (VOCs)) or oxides of carbon, nitrogen and sulphur (CNS) and particles (e.g. metals, soot carbon black, particulate matter (PM) and so on). It has thus been

reported that replacing fossil fuels with renewable energy could lessen untimely death rates, lost workdays, and moderate the costs for healthcare delivery (Weisser 2007; Patterson *et al.* 2016). By 2050, growth in world renewable energy consumption is anticipated to increase from 13 terawatts per year to about 30 terawatts (Kabir *et al.* 2018). In this regard, photovoltaic systems are potential devices that can convert solar energy and contribute significantly to the world energy since 100 PW irradiated power hits the earth surface on a sunny day (Luque and Hegedus 2011).

In transparent conducting oxides, combination of high optical transparency in the visible region of e-m spectrum and high electrical conductivity are qualities not easily achieved. A band gap greater than 3.2 eV is required for optical transparency in order to prevent absorption of electromagnetic spectrum in the visible region. Doping of metal oxides become very difficult as a result of this (Hosono 2007). For instance, if a suitable band gap in the host material electronic band structure and an appropriate donor energy level for an n-type dopant species are designed for a host material band structure, the ability of the host material to receive such dopant must be taken into consideration (Xu and Schoonen 2000). By this assertion, an increase in the deposition time of copper doped tin oxide leads to increase in the density of states generated by the impurities and forms a continuum of states below the conduction band leading to narrowing of the band gap of the host oxide. As a result, Cu-SnO<sub>2</sub> thin film has been observed in this study as a candidate for transparent



contact electrode material in the fabrication of optoelectronic and energy storage devices.

## EXPERIMENTAL PROCEDURE

Two different samples of Cu-SnO<sub>2</sub> were prepared at different deposition time. The substrate used for this work is Indium Tin Oxide (ITO) coated glass of 20 mm × 15 mm × 0.7 mm dimension. A simple home-made two electrode cell was used for the growth of the films. The advantages of the home-made electrode cell include low cost, high purity product, low temperature growth and ease of tunability for desired stoichiometry by a simple adjustment of deposition factors (Chopra *et al.* 2004).

### Preparation of precursor and sample deposition

A solution precursor (electrolyte) was prepared from some common chemical reagents. 5.64 g of SnCl<sub>2</sub>·2H<sub>2</sub>O salt was dissolved in 250 ml of distilled water to form 0.1 M of SnCl<sub>2</sub> solution. KOH of 0.1 M was prepared by dissolving 1.40 g of potassium hydroxide pellet in 250 ml of distilled water. Also, Cu(NO<sub>3</sub>)<sub>2</sub> of 0.01 M was obtained by dissolving 0.24 g of Cu(NO<sub>3</sub>)<sub>2</sub>·5H<sub>2</sub>O in 100 ml of distilled water. All the solutions prepared were kept in different standard flasks at room temperature (300 K) for twenty-four hours for a complete dissolution. Before sample deposition, some ITO coated glasses were washed properly with detergent, which were subsequently cleaned in an ultrasonic bath that contained acetone and methanol separately. The substrates were later dried briefly in an oven at a temperature of 110°C. For the deposition of Cu-SnO<sub>2</sub> thin film, an electrolytic solution containing 20 ml of 0.1 M of SnCl<sub>2</sub>, 20 ml of 0.1 M of KOH and 5 ml of 0.01 M of Cu(NO<sub>3</sub>)<sub>2</sub> was poured into the electrolytic container. The counter electrode (graphite) and the working electrode (ITO substrate) were held firmly to the holders and connected to power source and digital multimeter to achieve stabilized power. A cathodic potential of 1.60 V was selected for running the experiment in potentiostatic mode with anodic current degrading with growth time. Two samples tagged A1 and A2 were obtained at growth periods of 10 and 20 minutes, respectively.

### Sample characterisation

After drying the deposited films at 120°C for 5 minutes, they were annealed at 350°C in air for 1 hour. Surface morphology and chemical composition were studied using Zeiss ultra plus field emission scanning electron microscopy (FE-SEM) attached with energy dispersive X-ray spectroscopy (EDX) facility. An XPERT-PRO X-ray diffractometer (PANalytical BV, Netherlands) with reflection geometry ranging from 2θ values of 10 to 90° and having a step size of 0.01° and also operating with a Co Kα radiation source (λ = 0.178901 nm) at 50 kV and 30 mA was used to obtain the X-ray diffraction patterns. Optical absorption as well as transmittance of deposited films were studied across the visible spectrum region by a double beam UV/VIS spectrophotometer. Electrical characterisation of deposited thin films was investigated

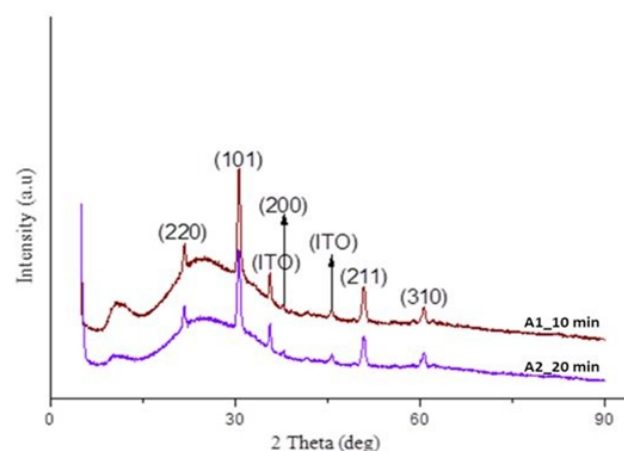
by a sheet resistance four probe station (T2001A1).

## RESULTS AND DISCUSSION

### Crystal structures and orientation

Figure 1 depicts the XRD pattern of Cu-SnO<sub>2</sub> thin film. Reflections along (220), (101), (200), (211) and (310) planes correspond to single phase of tetragonal rutile structure. Other observable peaks are attributed to ITO coated glass substrate (Jiang *et al.* 2008). Peak at the (101) plane is the most prominent, and it signifies a preferential growth orientation of the film particles. It was also observed that the intensity of the dominant peak (101) decreases with increase in deposition time. This suggests a slight deterioration in the crystallinity of SnO<sub>2</sub> structure, which could be attributed to the stress (contraction in the lattice due to copper [Cu<sup>2+</sup>: 0.73 Å] substituting some tin [Sn<sup>4+</sup>: 0.83 Å] sites) (Bagheri-Mohagheghi and Shokooh-Saremi 2004).

Across all the diffraction angles, several peaks due to Cu<sup>2+</sup> ions were not detected, this is probably due to a low percentage of the dopant, which agrees with some findings in literatures. It could even be due to some limitations from the XRD machine used especially its power ratings which may not be sufficient enough to identify the Cu particle (Choudhury *et al.* 2015; Benztouni *et al.* 2016). The reference card number for



**Figure 1:** XRD patterns of Cu-SnO<sub>2</sub> thin films at different deposition times.

the diffraction pattern obtained is with respect to JCPDS card number: 41-1445. Interplanar spacing, *d*, of the deposited thin film was determined by Equation.(1).

$$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \quad (1)$$

where *a* and *c* are lattice constants, and *h*, *k*, *l* are Miller indices. Williamson Hall's modified Debye Scherrer's formula in Equation 2 was used to determine the crystallites size of Cu-SnO<sub>2</sub> thin film (Gultekin *et al.* 2012):

$$\beta = \frac{k\lambda}{D\cos\theta} + 4\epsilon\tan\theta \quad (2)$$

where the constant  $\kappa$  is 0.94,  $\lambda$  is the wavelength of the incident X-ray,  $\beta$  is the full width at half maximum (FWHM) of the most prominent peaks, measured in radians and  $\theta$  is Bragg's diffraction angle and  $\epsilon$  is the residual lattice strain of the thin films. The intercept and slope of the plot of  $\beta \cos \theta$  versus  $\sin \theta$  were used to deduce the average crystal sizes and lattice strains of the samples respectively (Bagheri *et al.* 2013).

Dislocation density,  $\delta$ , was determined using the Equation (3) (Cullity and Stock 1956):

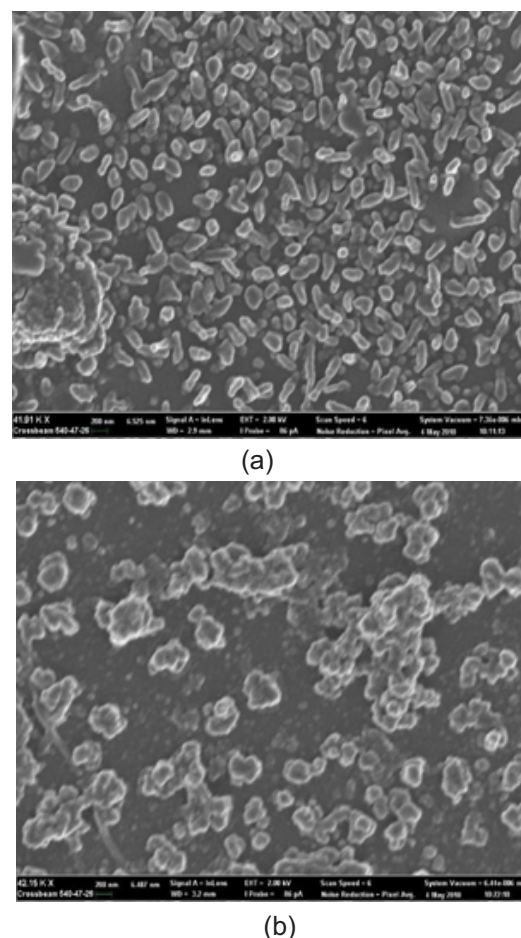
$$\delta = 1/d^2 \quad (3)$$

where  $d$  is the interplanar spacing.

Table 1 lists the value  $\epsilon$ ,  $D$ ,  $\delta$ ,  $a$ ,  $b$  and FWHM of the materials. From the table, it can be observed that the interplanar spacing  $d$ , crystallite size  $D$ , dislocation density  $\delta$  and lattice strain  $\epsilon$  of the material increased with increase in the dopant concentration. The increase in these properties shows that the increase in concentration of impurities can influence the crystal properties of electrodeposited nanostructures.

### Surface morphology

Figure 2 (a and b) shows the SEM micrographs of Cu-SnO<sub>2</sub> thin films. The particles are evenly distributed across the substrate, some of these particles are isolated, and some drift to form an agglomeration of particles exhibiting polycrystallinity. A distinct rice-like structure is observed on sample A1, such structure can allow easy passage of photons to proximate layer in photovoltaic device architecture. As the deposition time increases, less distinct but bigger dome-like



**Figure 2:** Surface morphology of the Cu-SnO<sub>2</sub> thin films at different deposition time for (a) A1 at 10 min and (b) A2 at 20 min.

**Table 1:** Calculated data from XRD pattern with  $a$  and  $b$  as lattice constants

| Sample | 2     | hkl | FWHM   | Lattice Constant |       | $d$ (Å) | $D$ (nm) | $(nm)^2$ | $\epsilon$ |
|--------|-------|-----|--------|------------------|-------|---------|----------|----------|------------|
|        |       |     |        | $a$              | $b$   |         |          |          |            |
| A1     | 30.68 | 101 | 0.3486 | 4.821            | 3.352 | 2.91    | 18.89    | 5.87     | 0.169      |
| A2     | 30.45 | 101 | 0.5821 | 4.813            | 3.341 | 2.95    | 21.61    | 16.40    | 0.277      |

particles with apparent surface defects that could be traced to stress emerged. Also, the image analyses revealed the particle size of sample A1 in nanometer scale (~15 nm) but at higher deposition time, the nanoparticles agglomerated to form cluster of particles and therefore enhanced particle sizes in samples A2 (~28 nm). In addition, the post-deposition annealing improves the crystallinity of the deposited layer. The absence of cracks in all the images could suggest that Cu ions (dopant) are well incorporated in the lattice of the host material (Zhang *et al.* 2013).

### Films Composition

The deposited samples were quantitatively analyzed by Energy Dispersive Spectroscopy (EDS). The spectra obtained is shown in Figure 3. The spectra reveal the presence of Sn and O along with some quantities of

copper. Other spectra observed are due to other elements present in the substrate and impurity atoms. The percentage atomic weight of the grown Cu-Sn-O and other elements due to the substrate are presented in Table 2.

### Electrical Characterisation

The electrical characterisation of deposited thin films was investigated by sheet resistant probe station (T2001A1). The result of the current-voltage measurement obtained for ITO and the deposited samples are presented in Figure 4(a-c). The plots revealed that the samples are ohmic conductor. The resistivity, conductivity and sheet resistance of ITO, and samples at different deposition time were determined by Equations (4) to (6).

$$\rho = R_s t_p \quad (4)$$

where  $\rho$  is the resistivity,  $R_s$  is the sheet resistance and  $t_p$

$$\sigma = 1/\rho \quad (5)$$

where  $\sigma$  is the conductivity and  $\rho$  is the resistivity.

$$R_s = 4.53V/I \quad (6)$$

$R_s$  is the sheet resistance, the applied inner voltage is denoted by  $V$ , and  $I$  the outer current. The results from the above equations are presented in Table 3. From the results, it is observed that sample A2 with higher deposition time has higher conductivity and lower sheet resistance than sample A1. This suggests its suitability as a transparent contact electrode for optoelectronic applications.

### Optical characterisation

Optical properties of the films were studied across UV/VIS range. The transmittance and absorption coefficient curves are presented in Figure 5. All the samples exhibited high absorbance in ultraviolet region. Absorbance drops from visible to infrared region with an occurrence of red shift in spectra presented in Figure 5a. Sample deposited at longer time absorbs better, this could be as a result of the thickness of the film. Absorption coefficient varies with photon energy at the optical absorption edge, this is due to electron excitation to higher energy level. Some metal oxides with such absorption band are used in organic

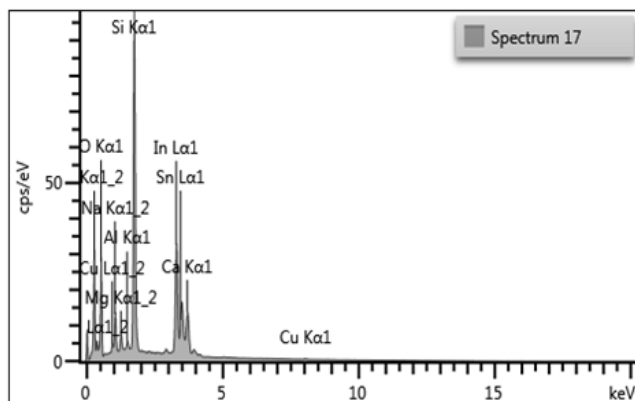


Figure 3: EDX elemental analysis of deposited Cu-SnO<sub>2</sub> thin film.

Table 2: EDX elemental analysis of electro-deposited Cu-SnO<sub>2</sub> thin film

| Element | wt%    | Atomic % |
|---------|--------|----------|
| O       | 23.17  | 48.11    |
| Na      | 3.68   | 5.32     |
| Mg      | 1.22   | 1.67     |
| Al      | 0.57   | 0.71     |
| Si      | 23.44  | 27.73    |
| Ca      | 4.11   | 3.41     |
| Cu      | 1.81   | 0.95     |
| In      | 37.08  | 10.73    |
| Sn      | 4.92   | 1.38     |
| Total   | 100.00 | 100.00   |

Table 3: Electrical measurement of blank indium tin oxide (ITO) and Electrodeposited Cu-SnO<sub>2</sub> Samples

| S/N | Sample | Conductivity (Ωm) <sup>-1</sup> | Resistivity (Ωm)      | R <sub>Sheet</sub> (Ω) |
|-----|--------|---------------------------------|-----------------------|------------------------|
| 1   | ITO    | $7.869 \times 10^5$             | $1.26 \times 10^{-6}$ | 12.70                  |
| 2   | A1     | $9.427 \times 10^5$             | $1.06 \times 10^{-6}$ | 1060.79                |
| 3   | A2     | $1.438 \times 10^5$             | $6.95 \times 10^{-6}$ | 69.53                  |

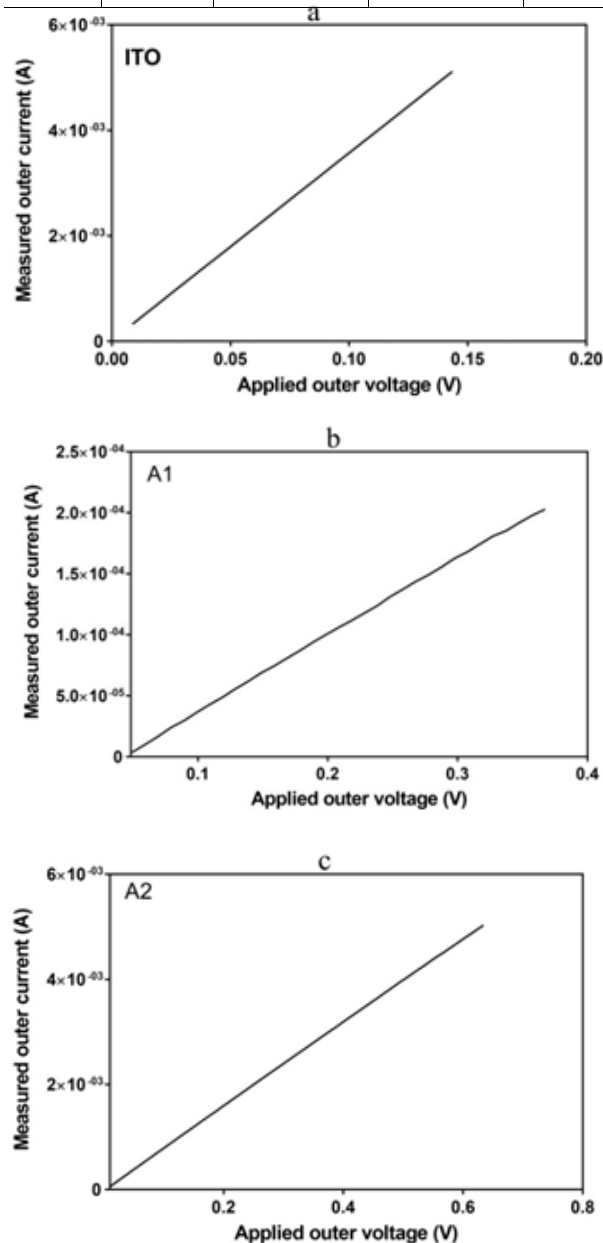
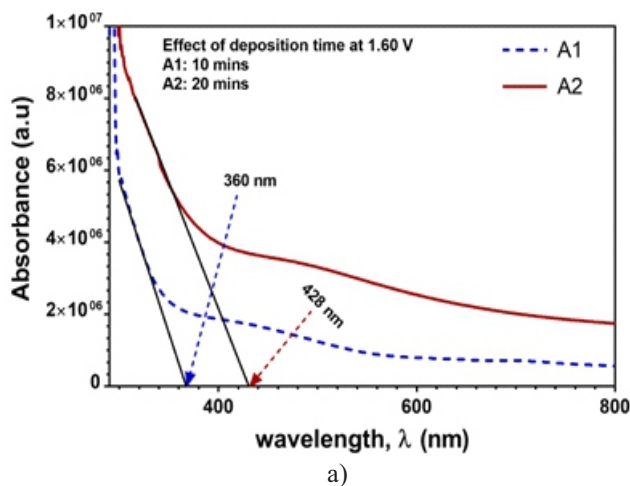


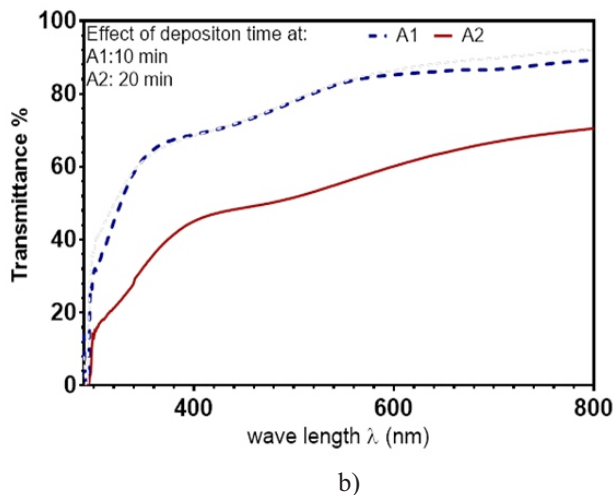
Figure 4: Plots of ITO and the Cu-SnO<sub>2</sub> thin film deposited at different deposition time (a) ITO (b) 10 min (c) 20 min.

photovoltaic cell as buffer layer (Benhaliliba *et al.* 2011). This implies that the deposited Cu-SnO<sub>2</sub> films can be used to make contact electrode capable of selectively absorbing photons across wide wavelength region and at the same time minimize recombination of photogenerated excitons. In Figure 5b, the Cu-SnO<sub>2</sub> samples obtained at shorter time exhibits better

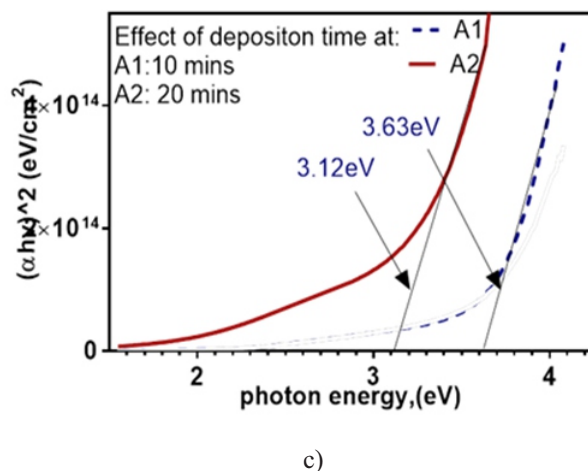




**Figure 5a:** UV-Visible absorption coefficient spectrum of deposited Cu-SnO<sub>2</sub> thin film. The absorption edges for A1 and A2 are 360 and 428 nm, respectively.



**Figure 5b:** UV-Visible transmittance spectrum of deposited Cu-SnO<sub>2</sub> thin film



**Figure 5c:** Tauc's plot for the estimation of energy band gap of Cu-SnO<sub>2</sub> thin film. The energy band gaps are A1 = 3.63 eV and A2 = 3.12 eV.

transmittance in the visible region. The absorption coefficient ( $\alpha$ ) could be calculated by the relation below (Guascito *et al.* 2008):

$$\alpha = 2.303 A/t \quad (8)$$

where A is the absorbance and t is the film thickness.

Tauc's plots in Figure 5c were generated from the measured absorption data. The energy band gaps were estimated in Equation. 9.

$$\alpha h\nu = B(h\nu - E_g)^n \quad (9)$$

where direct and indirect band gap are denoted by  $n = 1$  and 4 respectively.  $\alpha$  is the absorption coefficient (Anthony *et al.* 2007; Pradhan and Leung 2008), B is an empirical constant,  $h\nu$  is the photon energy and  $E_g$  represents the energy band gap. The energy band gaps of the samples were estimated by extrapolating the linear portion of  $(\alpha h\nu)^2$  values plotted against  $h\nu$  axis at  $(\alpha h\nu)^2 = 0$  (Jones and Lettington 1972; Márquezet *et al.* 1992). It was observed that the optical band gap decreased with longer deposition time. Spectrum obtained from sample A2 deposited at longer deposition time manifested lower optical band gap compared to that of A1 obtained at shorter times. This could be attributed to the thickness of the film. This lower energy band gap is specifically due to the addition of donor energy level by impurities, into the band gap of SnO<sub>2</sub>, which shifts Fermi level closer to the band edges (Xu and Schoonen 2000).

With increase in the deposition time of copper doped tin oxide, the density of states generated by impurities increased and formed a continuum of states below the conduction bands leading to reduction in band gap. Consequently, there can be higher charge mobility and reduced rate of surface recombination.

Figure 5c presents a plot of skin depth against photon energy. It can be observed from the plot that the skin depth decreased with photon energy. The spectra also revealed that sample A1 demonstrated higher skin depth than sample A2. The behaviour of the spectra revealed that skin depth was transmittance-related.

## CONCLUSION

In this study, Cu-SnO<sub>2</sub> thin film was deposited successfully on ITO coated glass using the electrodeposition technique. XRD pattern of the film was characterised with multiple peaks implying that the film is polycrystalline, with tetragonal structure. Morphological studies revealed compact, uniform and well distributed films, across the surface of the substrate. Polycrystals are formed by grain agglomeration and post-deposition annealing improved film crystallinity. EDX spectra revealed peaks that correspond to elements and compositions of the films and substrates. Other peaks are attributed to impurities. The deposited samples revealed ohmic properties by electrical characterisation. The optical absorption edges were determined between 360 and 428 nm for the samples respectively. Estimated direct energy gaps were 3.12 and 3.63 eV for the samples A1 and A2. This suggested that the deposited films can function efficiently as a

transparent contact electrode in optoelectronic devices.

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