Successive Ionic Layer and Absorption Reaction (SILAR): A synthesis technique of optimising the properties of zinc oxide/silver (Zno-Ag) nanocomposite thin films

Jayeoba[1*], B. O. and Olowofila[2], I. O.

[1]Department Science Laboratory Technology, Federal Polytechnic, Ilaro

[2]Department Science Laboratory Technology, Federal Polytechnic, Ilaro

Phone No: +234 8065633250
e-mail: jaybabpeterson@yahoo.com

Abstract

The efficiency of ZnO thin film can be enhanced for several applications by incorporating plasmonic silver nanoparticles through successive ionic layer adsorption reaction (SILAR) to form nanocomposite materials. This study uses Sol-gel method to synthesise ZnO and uses SILAR technique to fabricate ZnO-Ag composite material by varying the SILAR (cycles: 0, 2, 3, 4). The optical and morphological properties were characterised by surface profilometry, Scanning Electron Microscope, UV-vis spectrophotometer. The SEM micrograph shows that the film homogeneity and smoothness increases with increase in SILAR cycles of silver nanoparticles as structural defect or possible impurities in reaction components tend to disappear at higher SILAR cycle by successive accumulation of silver nanoparticles due to improved film morphology. The film thicknesses were found to linearly increase with SILAR cycle, and logarithmic decrease of the absorbance over wavelength 350-900nm and decrease in energy gap from 3.22eV for ZnO to 2.49eV for ZnO-Ag with 4 SILAR cycles were observed, these are results of successive deposition of the particles on the substrate and presence of p-type conductivity in the silver-doped ZnO nanoparticles which is responsible for the dominance of silver in samples property dictation. This study opens way to SILAR technique as a notable way of tuning the properties of nanocomposite materials.

INTRODUCTION

Background to the Study

Nanocomposite materials especially in its hybrid form possess exceptional properties and multifunctionalities. This has drawn the attention of researchers towards its applications in various field of study as it gives chance for tuning which cannot be achieved by single-component materials (Chu and Seeger, 2017). This hybridization that provides room for manipulation for improvement can be explored by trying a unique means of synthesis to fine-tune and optimize the functionalities in the field of application. This study consider successive ionic layer adsorption reaction as a secondary method to hybridize/synthesize nanocomposite zinc oxide- silver (ZnO-Ag) thin film, after the primary method of Sol-gel have been utilised to fabricate the single component material of zinc oxide (ZnO). This method of deposition is analogous to a modified chemical bath deposition (CBD) in which the thickness of the layer has been determined by the number of deposition cycles (Ashby, Shercliff, and Cebon, 2007). SILAR method involves the alternative immersion of the substrate in a solution containing a soluble salt of cation and anion of compounds to be grown (Sanjeev, Gajanan, Deuk, Dae-Young, and Thakur, 2018). Hence, the substrate supporting the growing film is rinsed in high purity deionized water after each immersion to avoid homogenous precipitation.

A two-step synthesis technique is proposed to a metal oxide semiconductor thin films zinc oxide (ZnO) because of its incredibly low cost, availability and good performance in high temperature environment with inherent properties like large band gap, higher electron mobility as well as high breakdown field strength which are highly required in most optoelectronic devices (Kumar, Kumar, Al-Dossary, and Umar, 2015; Isah, Ramalan, Ahmadu, Ibrahim, Yabagi, and Jolayemi, 2016).

Zinc oxide (ZnO) is one of the most promising and multifunctional semiconductor materials with a wide band gap, this material exhibit some fascinating physical, chemical and optoelectronic properties that suits several applications such as ultra violet (UV) electronics, spintronic devices, solar cell technology and sensors (Isah *et al.*, 2016; Jagadish and Pearton, 2006). However, the large band gap semiconductors suffer a drawback of limited light harvesting ability and recombination of electron and holes which can be minimised with the introduction of metal nanoparticles (Habibi and Sheibani, 2010), and therefore this study has proposed a unique method to tackle this problem by incorporation of plasmonic metal nanoparticles in ZnO thin film to finetune and optimize the properties ZnO-Ag nanocomposite

material through Successive Ionic Layer and Absorption Reaction (SILAR) Method. This method of nanocomposite incorporation will also overcome carriers trapping as well enhance the photocatalytic efficiency as a result of localised surface plasmons (LSP) effect of these metals. Therefore, this study aim to investigate effect of SILAR cycle variation on properties of plasmonic silver nanoparticles (AgNPs) incorporated zinc oxide (ZnO) thin films. And the following objectives would be carried out to achieve the aim by Synthesising ZnO thin films using Sol-Gel method; Incorporating AgNPs into the ZnO thin films via Successive Ionic Layer and Adsorption Reaction (SILAR) Method; and Investigation of the optical properties as well as morphological structure of the synthesised AgNPs embedded ZnO thin films would be carried out.

LITERATURE

Thin Films and Incorporation of Plasmonic Metal Nanoparticles

There is a wide variety of industrial applications of thin films which have great importance. Some of such applications include sensors, solar cells, wear-resistant coatings and corrosion-resistant coatings, actuators and porous nanacomposite films (Kumar *et al* 2015).

Noble metal nanoparticles including gold (Au), platinum (Pt), palladium (Pd) and silver (Ag) have been incorporated in wide-gab metal oxide semiconductors such as ZnO to overcome carriers trapping as well enhance the photocatalytic efficiency as a result of localised surface plasmons (LSP) effect of these metals (Sun, Qiao, Tan, Wang, & Qiu, 2012; Koleva *et al.*, 2013; Habibi and Sheibani, 2010; Koleva *et al.*, 2014). ZnO-Ag nanocomposite thin films is increasingly attracting a considerable attention due to its low cost and its excellent tuneable structural and optoelectronic properties to match with specific application by controlling synthesis parameters and process (Isah *et al.*, 2016; Liu, Bao, Yang, Lu, Cheng, Qu, . . . Zhang, 2014; Segets *et al.*, 2009). Several methods including photoreduction, pulsed laser deposition, hydrothermal, nonionic polymer assisted thermolysis, microwave-assisted synthesis and sol-gel methods have been reported to synthesise ZnO-Ag nanocomposite thin films (Sun *et al.*, 2012), many of these techniques are however complicated requiring a special condition like high temperature or low pressure environment as well require a long reaction time. Besides, some of these methods also make use of toxic components and involve expensive experimental setup.

Successive Ionic Layer and Absorption Reaction (SILAR) Method

SILAR, a name ascribed by Nicolau *et al.* (1988), stands for Successive Ionic Layer Absorption and Reaction. SILAR method involves the alternative immersion of the substrate in a solution containing a soluble salt of cation and anion of compounds to be grown. Hence, the substrate supporting the growing film is rinsed in high purity deionized water after each immersion to avoid homogenous precipitation. Summarily, SILAR is a method of deposition in which the thickness of the layer has been determined by the number of deposition cycles.

SILAR method is often referred to as modified Chemical Bath Deposition (CBD). Deposition of thin films occurs in CBD due to substrate maintained in contact with dilute chemical bath – the film formation on substrate in CBD occurs when Ionic Product (IP) exceeds Solubility Product (SP). But in SILAR method, thin films are developed by immersing the substrate into cationic and anionic precursors which are placed separately to get reactions at temperatures which are chosen. It is rinsed in deionized water before the next immersion and very important in SILAR is the rinsing time mainly for ionic layer formation (Jagadish & Pearton, 2006).

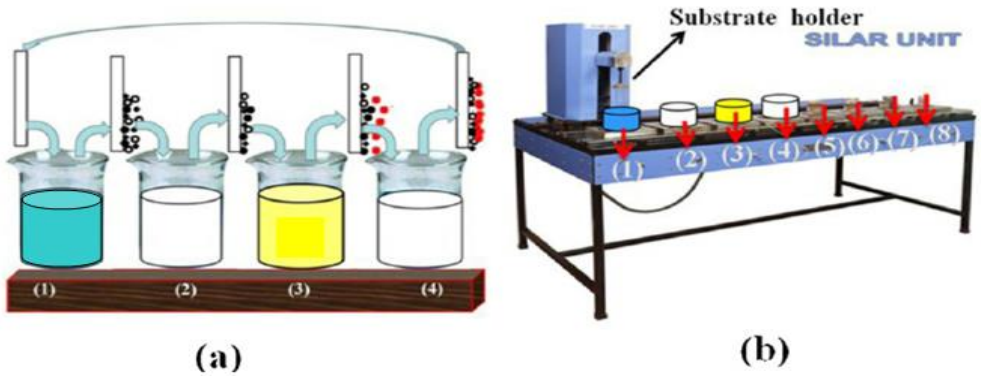

Figure 1: A Typical SILAR Unit (Jagadish & Pearton, 2006)

MATERIALS AND METHODS

Materials

The materials that are used for the synthesis of ZnO-Ag nanocomposite thin films are Reagents/Chemicals, Glassware and apparatus, and Electrical appliances and devices. The reagents are analytical, according to Molarities/dimension, the solution prepare from this study with reagents has three classes: Piranha solution, Precursor and SILAR solution.

The Reagents/Chemicals includes: Silver nitrate ($AgNO_3$), Stannous chloride ($SnCl_2.2H_2O$), Ammonium solution (NH_3OH) (*ca.* 33 %wt NH), Hydrochloric acid (HCl) (36%), Ethanol (99

%), Zinc Acetate ($Zn(O_2CCH_3)_2(H_2O)_2$), Cetrimonium bromide (CTAB),Sulphuric Acid (H_2SO_4), Hydrogen peroxide (H_2O_2), Soda Line Rulers (microscope slide), Distilled water.

Methods

Preparation of Piranha solution which is a mixture of 98% of sulfuric acid (H_2SO_4) and 30% hydrogen peroxide (H_2O_2) was carried out to wash the soda line rulers (microscope slides).

Then, precursor solution of (ZnO NP) is synthesised through a process called Sol-gel. Where 5.5 grams of Zinc Acetate (ZnAc) was weighed out into a beaker, 20 ml of Centrimonium bromide (CTAB) was added as a solvent and 20 ml of ammonia solution (NH_3OH) was added as a stabilizer, propanol was added to the solution until it reaches 70ml of the beaker. Then the solution was placed on a magnetic stirrer, it was stirred for 3 hours to get a clearer solution and the zinc oxide will polymerize to form a gel and it is orange in colour.

Next is the SILAR solution**,** this consists of four solutions which are; Sn^{2-} solution (cation) for adsorption, distilled water (H_2O) for rinsing, Ag^{+} solution (anion) for reaction and distilled water (H_2O) for rinsing.

Preparation of ZnO and ZnO-Ag Samples (SILAR)

A two-step sequential deposition method was used to synthesise ZnO-Ag nanocomposite involving growing ZnO thin films on glass substrates via sol-gel method and subsequently depositing a layer of Ag NPs using successive ionic layer adsorption and reaction (SILAR) method.

Preparation Slides for Sample Deposition

The Piranha solution was poured into a beaker up to 80ml, and then four (4) slides were load up into the Piranha solution, the solution boils for 7 minutes after which the slides were removed from the Piranha solution with a spatula into a plastic bowl filled with distilled water. This was done to wash the slides and preserve them from contamination. The glass substrates were prepared which was first cleaned/degreased in a polar solution containing Sodium Lauryl, rinsed with distilled water, ethanol and dried under compressed hot air for 5 minutes.

Synthesis of ZnO

The first slide was pick out from the distilled water with a spatula and was attached to the clip of the stepper motor, then the stepper motor was stated from the program on the computer to enable the slides down into the precursor which is ZnO, after which the slides was detached from the stepper motor and then flipped to avoid dripping when drying. The slide was dried

with a blower and then transferred on the plate of the magnetic stirrer hotplate to heat it up with the temperature of 170^{o}C for 15 minutes, later moved on the electric hot plate to heat it with the temperature of 450^{o}C for 2 hours, after 2 hours the slide was removed from the hotplate, it was allow to cool down before it was labeled ZnO only.

Synthesis of ZnO with Variation of SILAR Cycle

The first step is applied to a second slide, after which a SILAR setup was prepared, four beakers was needed, the first beaker contains Sn^{2-} solution, the second beaker contains distilled water, the third beaker contains Ag^{+} and the fourth beaker contains distilled water.

The slide which now has a layer of ZnO was put into the Sn^{2-} solution for 2 minutes after which it was moved into the distilled water for rinsing and moved into the Ag^{+} for another 2 minutes and then moved into the last beaker filled with distilled water for rinsing, the slide now complete 1 SILAR cycle, so we repeat the process to complete 2 SILAR cycle. The slide was dried up with the blower and moved on the magnetic stirrer hotplate to heat it up for 10 minutes and later transferred onto the electric hotplate to heat up for 1 hour and 30 minutes, then we now have a slide with ZnO and 2 SILAR cycle.

The above processes were repeated for the fabrication of thin film with 3, 4 and 5 SILAR cycles respectively. This gives rise to four (4) different samples as tabulated in the Table 1.

Table 1: Composition and SILAR Cycle Summary of Samples

Samples	Composition	SILAR Cycle
A	ZnO	0
B	ZnO/Ag	2
C	ZnO/Ag	3
D	ZnO/Ag	4

RESULTS AND DISCUSSION

After the synthesis of the Zno-Ag sample, the prepared samples were characterised using variour characterisation techniques to investigate the properties of each. The film thicknesses were evaluated from the profilometer roughness data and spectral and the results are summarised in Table 2) as a function of SILAR cycles.

Table 2: Summary of the Thickness ZnO and ZnO/Ag Thin Film at Different SILAR Cycles

Samples	**Thickness (µm)**
ZnO	0.25
ZnO/Ag at 2cycles	0.34
ZnO/Ag at 3cycles	0.41
ZnO/Ag at 4cycles	0.49

From Table (2) it was observed that the film thickness increased linearly with an increase in the number of cycles, representing a typical characteristic of the sol-gel spin coating technique. This linear increment behavior of thickness of ZnO and ZnO/Ag could be attributed to the adhesion or accumulation of successive formation of new layer by the introduction of silver nanoparticles as the SILAR cycles increase. This result can be further verify from the observed effect of the thickness on the fabricated samples of ZnO and ZnO/Ag thin film since Vinod Kumar, Neetu Singh. Mehra, Avinashi, Purohit, & Swart. (2013). suggests that as the film becomes thicker, the crystalline quality of the films is improved and the preferred orientation along the ***c-axis*** can be observed.

Optical Characteristics: Absorbance Relation of ZnO and ZnO/Ag Thin Film

The major optical data acquired from the UV–vis spectrophotometer is the absorbance-wavelength values within the visible range as indicated by the absorbance edge at 350 *nm* in Figure 2. From this relation, other optical parameters are estimated in this section.

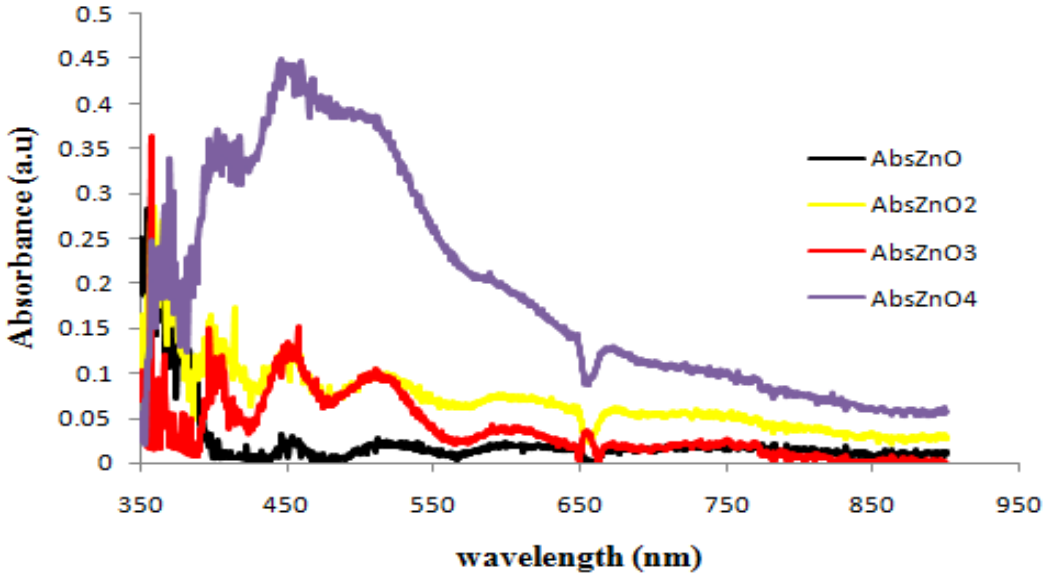

Figure 2: Absorbance Wavelength Variation of ZnO and ZnO/Ag Thin Film as a Function of SILAR Cycles

The curves produced by the UV-vis Absorbance data were observed to be in agreement with Absorbance relation within the visible range of wavelength. The more a particular wavelength of light is absorbed by a substance, the less it is transmitted. It was observed from the spectral that the sample with highest number of SILAR cycles has relatively highest absorbance compare to the samples with smaller number of SILAR cycles; this logarithmic increase of the absorbance with increment in SILAR cycles is attributed to the accumulation of silver nanoparticles as a result of successive deposition of the particles on the substrate. Optical absorption measurements indicated shift in the absorption band edge by silver doping with increment in SILAR cycles. Due to the high electronegativity of Ag than Zn, electron transfer was assumed to occur from Zn to the Ag particles, suggesting that the chemical bond between ZnO and Ag is one reason for silver effect in controlling defects at ZnO crystal lattice Hosseini *et al.* (2015).

Absorption and Extinction Coefficient SILAR Relation of ZnO and ZnO/Ag Thin Film

Absorption and extinction coefficients are important optical parameter of thin film. Figure 2 shows the coefficient as a function of SILAR cycles for ZnO and ZnO/Ag using Lambert-Beer law (Islam and podder, 2009):

$$\alpha(\lambda) = \frac{2.303A}{d} \qquad (1)$$

where *d* is the sample thickness, where *A* is the absorbance. The extinction coefficients are calculated from the following relation (Pankove, 1971):

$$k(\lambda) = \frac{\alpha\lambda}{4\pi} \qquad (2)$$

The fraction of incident radiant energy absorbed per unit thickness, *d* of the thin film of ZnO and ZnO/Ag are plotted below:

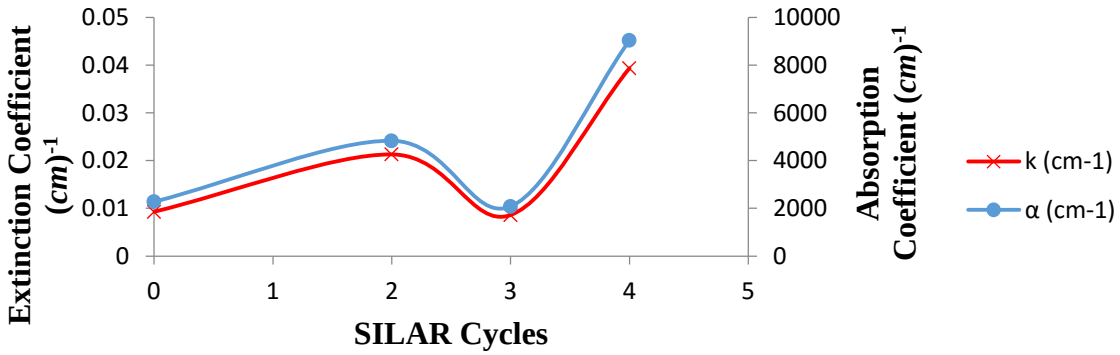

Figure 3: Absorption and Extinction Coefficient Comparison with SILAR Cycle

Since Absorption and extinction coefficients are a measure of the rate of decrease in the intensity of electromagnetic radiation (light) as it passes through a given substance; the fraction of incident radiant energy absorbed per unit thickness of ZnO and ZnO/Ag as the SILAR cycle varies. The trends shown above are indication of the absorption capability of the deposited films of ZnO and subsequent introduction of Silver nanoparticles in a successive immersion. The coefficients tend to rise linearly with increase in SILAR cycles but suddenly depict low absorption coefficient at 3cycle this is due to absorbance variation with wavelength, The high value of absorption coefficient obtained for the film deposited at 4 indicates high absorption and reduced transmittance as compared to films deposited at other number of SILAR cycle.

Optical Bandgap of ZnO and ZnO/Ag

From solid state band theory, the relation between the optical absorption and the energy of the incident light *hv* is given by Tauc's and Davis–Mott in relation in Equation (3)

$$\alpha = \frac{A(hv - E_g)^n}{hv} \qquad (3)$$

The optical energy gap of the ZnO and plasma nanoparticles of ZnO/Ag silver was obtained from drawing the graph of $(\alpha hv)^2$ versus energy, according to Equation (3). In this relation, A is absorption constant, *h* is Planck's constant, *v* is light frequency, *n* = 1 for direct electronic

transition, n = 4 for indirect electron transition and E_g is energy gap (Whang, Hsieh, Chen, 2012).

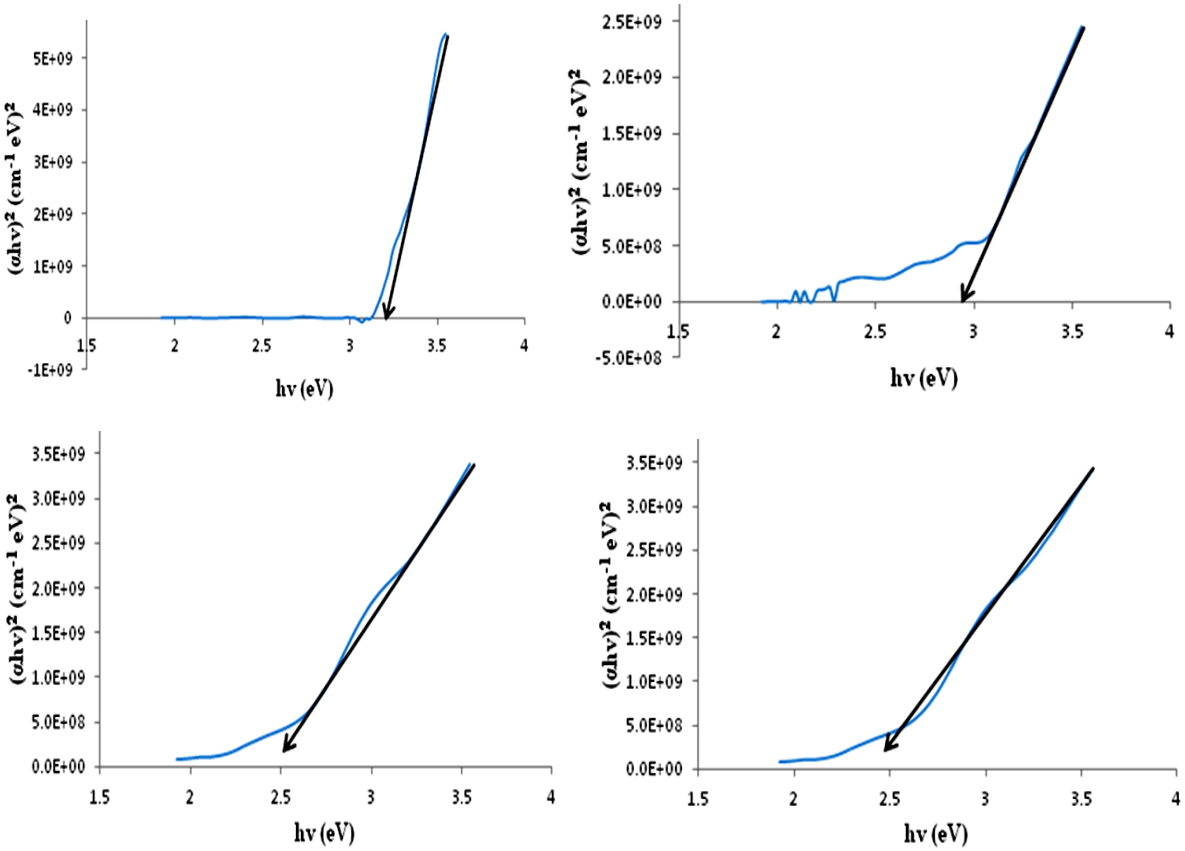

Figure 4: Extrapolation of Optical Bandgap for (a)ZnO (b) ZnO/Ag at 2 Cycles (c) ZnO/Ag at 3 Cycles (d) ZnO/Ag at 4 Cycles

Table 3: Summary of Optical Bandgap

Sample	SILAR Cycle	Bandgap (*eV*)
ZnO	0	3.22
ZnO/Ag at 2 Cycle	2	2.91
ZnO/Ag at 3 Cycle	3	2.62
ZnO/Ag at 4 Cycle	4	2.49

Figure and Table 3 indicates the optical bandgap of the samples (ZnO and ZnO/Ag) at different SILAR cycle as obtained from the UV-vis data after extrapolation of Tauc's plot. The ZnO sample without silver nanoparticles has the highest sample's optical energy gap of 3.22 eV. The optical bandgap decreases linearly with increase in SILAR cycles. With increasing number of SILAR cycles of silver nanoparticles, the energy gap decreased from 3.22 eV for the pure sample (ZnO) to 2.49 eV for the sample ZnO/Ag at 4 cycles, as shown in Table 3.

The increase of the energy gap for ZnO doped with donor is commonly observed, while reducing the energy gap of ZnO doped with acceptor has been reported (Gupta *et al.*, 2011). Decreasing the energy gap can be related to the presence of p-type conductivity in the silver-doped ZnO nanoparticles. Silver doping in ZnO provides the impurity band in the energy gap, which could be due to the formation of the p-type in this substance. It should be mentioned that this reduction in energy gap led to increase efficiency in the use of these materials in optoelectronic devices.

Morphological Characteristics of ZnO and ZnO/Ag: SEM

The surface morphology of the samples were investigated using Scanning Electron Microscope at STEP B and the micrographs are displayed in the Figures 5, the texture, roughness and grain size can be observed.

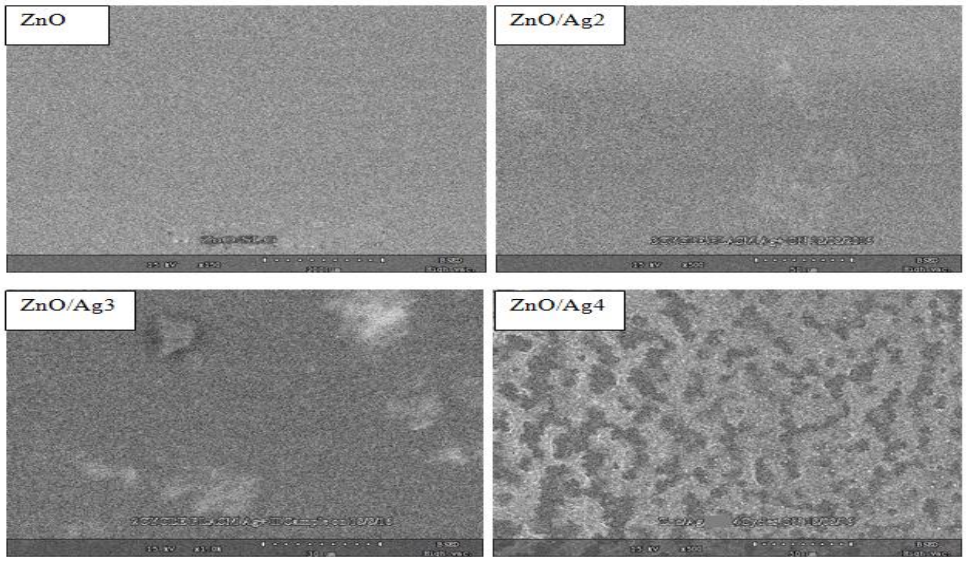

Figure 5: SEM Micrograph for (a)ZnO (b) ZnO/Ag at 2 Cycles (c) ZnO/Ag at 3 Cycles (d) ZnO/Ag at 4 Cycles

The SEM micrograph shows that the film homogeneity and smoothness increases with increase the SILAR cycles of silver nanoparticles. With an exception in the film with 2 SILAR cycle, that sample have a porous and uneven surface leading to rough film surface and poor surface morphology of deposited films relative to film with 3 and 4 cycle. While films deposited at 4 SILAR tends to show an improvement in the structure and enhancement in the film uniformity. By increasing the SILAR cycle from 2 to 4, the surface gets fully covered with fine and smooth grains which lead to better surface morphology and crystallographic structure. This could be attributed to possible structural defect in deposited film or possible impurities in reaction components which tend to disappear at higher SILAR cycle successive

accumulation silver nanoparticles due to improved film morphology. And this is due to the high electronegativity of Ag than Zn, electron transfer was assumed to occur from Zn to the Ag particles, suggesting that the chemical bond between ZnO and Ag is one reason for silver effect in controlling defects at ZnO crystal lattice Hosseini *et al.* (2015). These results can be confirmed with crystallographic investigation using XRD.

CONCLUSION

Conclusion

In conclusion, this study has synthesized, characterized and investigated the optical and morphological properties of ZnO and ZnO/Ag nanoparticle thin film prepared by Sol-gel method and the SILAR cycles are varied for four (4) samples.

Film thickness was found to increase with increase in SILAR cycles. This thickness variation is attributed to increment in the successive accumulation of silver nanoparticles layer as the SILAR cycle is increased, therefore film with 4 SILAR cycle recorded highest value of thickness of 0.49 μm. The thickness values were used in calculating some important optical parameters like absorption and extinction coefficients, this means that if other variables in these coefficients remain constant then thickness plays a crucial role in the investigation of the sample optical properties.

UV-vis spectrophotometer was used to characterise the samples for optical properties investigation. Transmittance, absorbance, absorption coefficient, extinction coefficient and optical bandgap were explored from the data and corresponding spectral within the visible range of 350 nm and 900 nm. Relatively, the film with highest number of SILAR cycle has the highest absorbance value with the pure ZnO produced with Sol-gel method has the absorption capability. Sample with 2 and 3 SILAR cycles has similar trends of absorbance but can be differentiated with intensity peaks from 510 nm - 900 nm. It could be concluded that as the ionic layers are increased, it gives room for accommodation of more silver nanoparticles that absorb electromagnetic radiation at different wavelength thereby increasing the absorbance of the sample.

Absorption and extinction coefficients of the fabricated samples are determined and the high value of absorption coefficient obtained for the film deposited at 4 SILAR cycles indicates high absorption and reduced transmittance as compared to films deposited at other number of SILAR cycle.

The optical bandgap decreases with increase in SILAR cycles from 3.22 eV to 2.49 eV an indication of the presence of p-type conductivity in the silver-doped ZnO nanoparticles. Silver

doping in ZnO provides the impurity band in the energy gap, which could be due to the formation of the p-type in this substance. It should be mentioned that this reduction in energy gap led to increase efficiency in the use of these materials in optoelectronic devices.

It is recommended that this method of synthesis should be adopted for fine tuning nanocomposite materials for application purposes and characterised for other properties investigation.

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