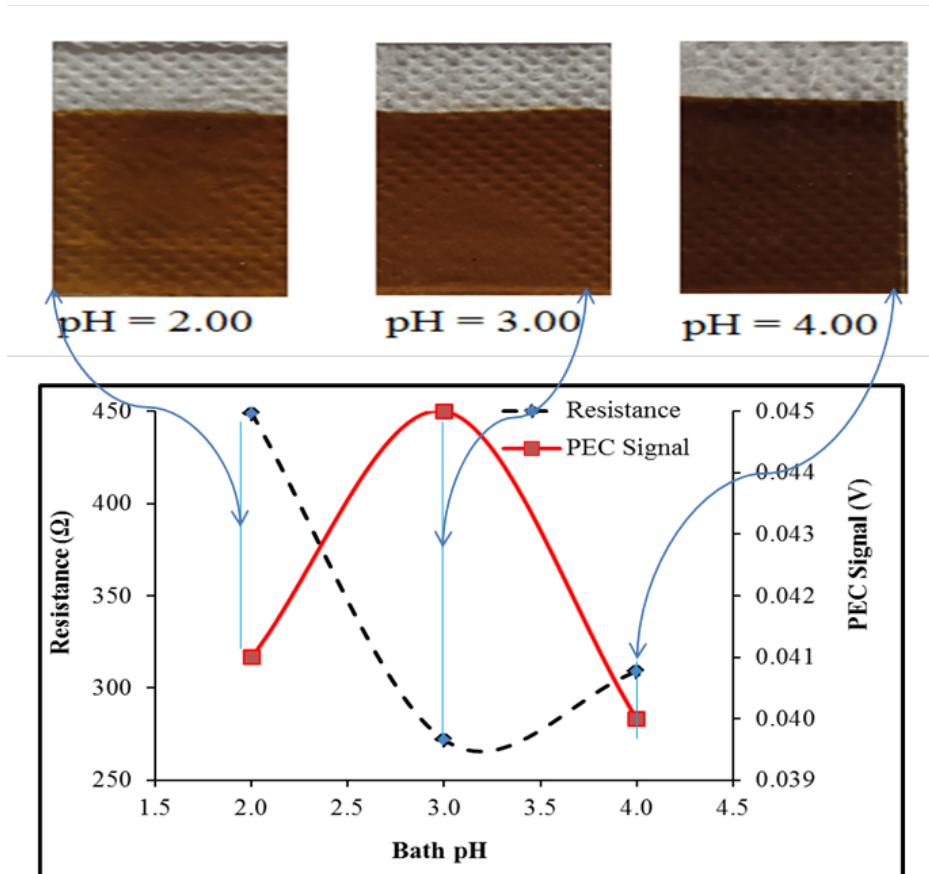


RESEARCH ARTICLE

Effect of pH variation on the optoelectronic properties of electrodeposited Al_2GaSe_3 ternary compound semiconductor

O. I. Olusola, J. O. Awodeyi, N. E. Adesiji, O. O. Olusola, O. A. Ajayi, S. B. Ibikunle and S. S. Oluyamo



Highlights

- Successful electrodeposition of Al_2GaSe_3 thin films in an acidic bath medium
- Electrolytic bath pH variation influences the material optoelectronic properties
- The visual appearance of Al_2GaSe_3 semiconductor changes with variation of bath pH
- All electrodeposited Al_2GaSe_3 layers show p – type electrical conduction

Effect of pH variation on the optoelectronic properties of electrodeposited Al_2GaSe_3 ternary compound semiconductor

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Abstract: The growth of aluminum gallium selenide (Al_2GaSe_3) ternary compound semiconductors has been achieved in this work using electrodeposition technique at different electrolytic bath pH values of 2.00, 3.00 and 4.00. The deposition was carried out using a microcontroller – based potentiostat with two-electrode electrochemical cell with the goal of attaining a semiconductor with improved electrical and optical properties. The aqueous electrolytic baths were prepared using 0.1 M aluminum chloride hexahydrate ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$) as Al^{3+} source, 0.1 M gallium chloride (GaCl_3) as Ga^{3+} source and 0.01 M SeO_2 as Se^{2-} source in 800 mL of deionised water. The bath temperature was maintained at 70 °C and a cathodic potential of 1200 mV was used for the thin films deposition. The electrical and optical properties of the electrodeposited Al_2GaSe_3 thin films were characterized using photo-electro-chemical (PEC) cell measurement and ultraviolet-visible spectrophotometer respectively. It was observed that the optical band gaps of the Al_2GaSe_3 thin films were pH dependent. The electrical conductivity types obtained were mainly p – type in electrical conduction with the highest PEC signal obtainable at pH of 3.00. Both optical and electrical results revealed pH of 3.00 as an optimum pH to electroplate Al_2GaSe_3 thin films.

Keywords: Electrodeposition; pH variation; Al_2GaSe_3 thin films; Electrical; Optical.

INTRODUCTION

Semiconducting materials are unique materials that contribute greatly to the present age electronics. Semiconductors have a resistivity that lies in – between that of an insulator and a conductor (Dharmadasa, 2013). They are substances which comprises of inadequate amount of conduction electrons at room temperature. Its ability to respond to conduction makes it a very essential material in electronic appliances (Rahman, 2014). Group III (gallium, aluminum, indium) and Group VI (selenium, tellurium) are materials that are involved in many technological applications especially in solar cells and have gotten a lot of attention in the research world. Like gallium arsenide (GaAs) and gallium nitride (GaN), they are functional in electronic components and used also in optoelectronics in

relation to infrared application. In a similar manner, gallium selenide (GaSe) has an existing possibility as non-linear optical material and great potential as photoconductor (Alvi, 2013). Moreover, aluminum selenide (Al_2Se_3) has been known to be a promising material because they have direct band gap energy, better charge transport, good absorption co-efficient and high transmittance (Olubosede *et al.*, 2020). As well, Al_2Se_3 has been said to be a good window layer material that is useful for the formation of hetero – junction together with absorber layer materials like lead sulfide and cadmium telluride (Faremi *et al.*, 2021). However, GaSe has weak mechanical properties, high dislocation density and possesses high equilibrium partial pressures of Se (Ni *et al.*, 2013). Thus the application of GaSe in real application is therefore encumbered. To further make GaSe more technologically useful, it is being deposited with aluminum in a view to check the effect of the bath pH variation on optoelectronic properties of the ternary compound Al_2GaSe_3 . Different techniques can be used for growing the Al_2GaSe_3 , such as vacuum evaporation, chemical bath deposition (CBD), electrodeposition, spray pyrolysis among many others. However, some of these growth techniques have used sophisticated and expensive equipment as well as complex mode of operation. Electrodeposition technique has been used in this work due to its cost effectiveness and simplicity of operation. Electrodeposition process is a popular technique which can be used to deposit various electronic materials for applications in nanotechnology and macroelectronic – based devices (Dharmadasa and Echendu, 2012). The electrodeposition technique has other advantages which include: ability of band gap engineering, low temperature growth, control of film thickness by deposition time and potential variation (Olusola *et al.*, 2016). In this work, Al_2GaSe_3 thin films were electrodeposited at different electrolytic bath pH ranging from 2.00 to 4.00 and the influence of electrolyte pH variation on the materials optoelectronic properties were examined.

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MATERIALS AND METHODS

FTO glass plate of 2.5×2.5 cm² in dimension was thoroughly cleaned with soap solution and water and then dipped in a methanol to further remove any greasy substance. Samples were rinsed also with deionised water before the commencement of deposition. After the whole cleaning process, it was attached to a carbon rod using polytetrafluoroethylene (PTFE) tape which served as the working electrode in the electrodeposition setup.

The precursors used for the growth of Al₂GaSe₃ thin films were 0.1 M of aluminum chloride hexa-hydrate (AlCl₃·6H₂O) as Al³⁺ source, 0.1 M of gallium chloride (GaCl₃) as Ga³⁺ source and 0.01 M of selenium dioxide (SeO₂) as selenium ion source. The salts were dissolved in a 1,000 mL glass beaker containing 800 mL of deionised water to make up the aqueous electrolyte. The pH of the ternary compound solutions was found to be 1.13 after the mixture; it was then adjusted to 2.00 by adding drops of diluted ammonia solution and the bath was heated to an adjusted temperature of 70 °C. Same process was adopted for the growth of films at the pH of 3.00 and 4.00.

Electrodeposition was done in a potentiostatic mode with the use of two-electrode system, this is mostly known for impurities reduction in the electrolytic bath and low cost (Olusola *et al.*, 2015). The components making up the system include; electrolyte, power supply, electric heater with a magnetic stirrer, electrodes comprising of the anode (this serves as the counter electrode) and cathode (this functions as the working electrode). The electrodes were both immersed in the electrolyte. The deposited films were achieved at a temperature of 70 °C and growth duration (t_g) of 15 minutes using three different bath pH values.

Experimental Techniques

The optical properties of the electrodeposited (ED) Al₂GaSe₃ such as energy band gap, transmittance, absorbance, reflectance, extinction and absorption coefficients were obtained with the aid of ultraviolet visible spectrophotometer in the wavelength range of 300 nm to 900 nm.

Photo-electro-chemical (PEC) cell measurements were performed to establish the electrical conductivity type of the ED-Al₂GaSe₃ layers. PEC cell is a simple technique used for the determination of electrical conductivity type of semiconductors. It includes a semiconductor and electrolyte with an interface connection between them. The set-up for the PEC cell measurement includes glass/FTO/semiconductor that actually serves as the semiconducting electrode, a carbon electrode which is used as the counter electrode and the electrolyte prepared from 0.10 M of sodium thiosulphate (Na₂S₂O₃) in a 100 mL of deionised water and Iso –Tech current – voltage (I-V) analyser. The semiconductor and counter electrodes were both connected to the negative and positive terminals of the Iso – Tech I-V analyser respectively with the aid of connecting probes. The PEC set – up requires the insertion of the two electrodes inside the electrolyte. The potential between the two electrodes are then measured under both dark and light conditions. The difference between the voltage measured

under dark (V_D) and light (V_L) gives the PEC signal. The Iso – Tech I – V analyser set – up was also used to obtain the thin film resistance.

RESULTS AND DISCUSSION

Effect of pH variation on the visual appearance of ED-Al₂GaSe₃ layers

The experiments carried out to study the effect of pH variation on the growth of Al₂GaSe₃ layers showed that ED-Al₂GaSe₃ semiconductors can be deposited within a pH window of 2.00 to 4.00. Figure 1 reveals the visual appearance of ED-Al₂GaSe₃ semiconductors; the experimental observations showed that the colour changes from brown to deep brown by gradual changing of the pH from more acidic to less acidic medium. The experimental results showed that uniform Al₂GaSe₃ layers can be grown in an acidic solution (lower pH) when using an electrodeposition technique. At the pH value of 2.00, the colour of the deposited thin film was observed to be brownish, which got slightly deepened at the pH value of 3.00. At the pH value of 4.00, the brownish colour of the thin film further deepens. This result revealed that variation in pH of the electrolytic bath influences the colour of the deposited layers.

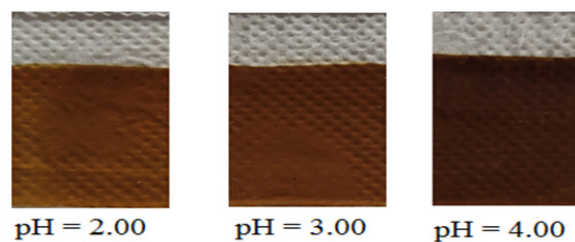


Figure 1: Visual appearance of electrodeposited Al₂GaSe₃ at varying pH values of 2.00, 3.00 and, 4.00. ($t_g = 15$ minutes, $V_g = 1200$ mV, $T = 70^\circ\text{C}$).

Effect of pH variation on the optical properties of ED-Al₂GaSe₃ layers

The different optical plots for the ED-Al₂GaSe₃ thin films are presented in Figures 2 – 6. Optical absorption measurements of the ED-Al₂GaSe₃ layers were carried out to estimate the optical energy band gap (E_g). It was revealed that the pH of the electrolytic solution has a significant effect on the band gap of the Al₂GaSe₃ thin films. The plot of $(ah\nu)^2$ against the photon energy for the Al₂GaSe₃ layers deposited at pH of 2.00, 3.00, and 4.00 of the electrolytic bath is shown in Figure 2. The straight line portion of the graph is extrapolated to the photon energy axis. The intercept on the photon energy axis gives the E_g value when $(ah\nu)^2$ is zero. The energy band gaps for the electrodeposited samples were found to be 2.65 eV for the film grown at pH of 4.00, 2.75 eV for the film deposited at pH of 3.00 and 2.90 eV for the Al₂GaSe₃ layers grown at pH of 2.00. At the pH of 2.00, the band gap was found to be the highest, indicating that it can be used as a window layer due to this high band gap and transparency. Likewise, there is the possibility of using the darker brownish layer

of Al_2GaSe_3 grown at the pH of 4.00 as an absorber layer in solar cell application if the deposition duration is increased beyond 15 minutes. Overall, the experimental results showed that the band gap reduces with increase in the pH of the electrolyte. The reduction of the band gap could be as a result of deposition of more aluminum and gallium metal ions at increased pH; another possible reason could be as a result of improvement of grain size of the films. The presence of chlorine and oxygen in the bath could also act as involuntary dopants in the electrolyte, which can have an effect on the materials opto – electronic properties (Sajeesh et al., 2012).

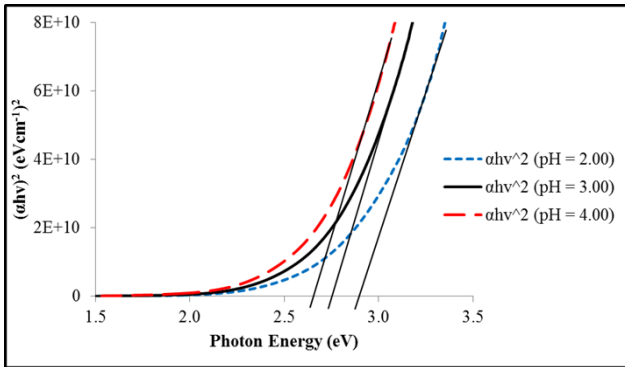


Figure 2: Energy band gap estimation of ED- Al_2GaSe_3 at different bath pH.

Figure 3 shows the graph of transmittance spectra of thin film layers with pH of 2.00, 3.00, and 4.00 in wavelength ranging from 300 to 900 nm. It was observed that the transmittance increased with increase in wavelength from the visible region to the near-infrared region of the spectrum. This denotes that the material has a good transmittance and will be a good window material during solar panel fabrication.

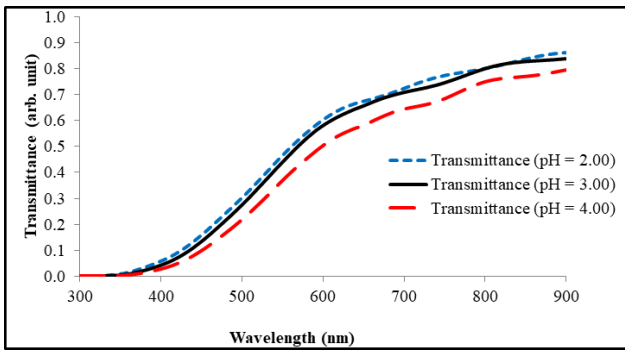


Figure 3: Plot of transmittance of ED- Al_2GaSe_3 films against wavelength at different bath pH.

Figure 4 shows the reflectance spectra of the thin films deposited at the pH of 2.00, 3.00, and 4.00. It was observed that the thin films have very low reflectance in contrast to their transmittance. At the wavelength of ~ 450 nm, the reflectance increased from 0.0 to 0.2 at barely ~ 500 nm and from there, the reflectance went on a sloppy decrease with increase in the incident photon wavelength from visible region to the near-infrared region. The low reflectance value shows that the films have very good potentials which can enable it to function as anti-reflection coating in solar

cells applications.

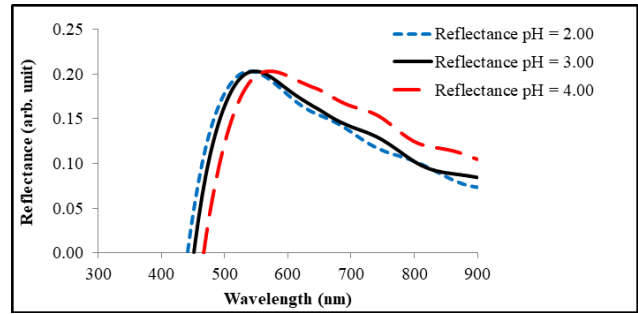


Figure 4: Plot of reflectance of ED- Al_2GaSe_3 films against wavelength at different bath pH.

Figure 5 shows the absorbance response of the Al_2GaSe_3 thin films. The absorbance reduces with increase in the wavelength of the incident radiation. Figure 5 illustrates that the thin films have the tendency to absorb high energy photons from the solar spectrum. The differences in the films absorbance are mostly conspicuous from the ultraviolet to the visible region. In the near end of the infrared region, all the films have nearly same absorbance.

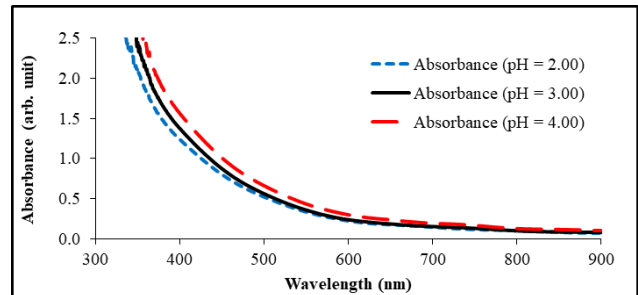


Figure 5: Plot of absorbance of ED- Al_2GaSe_3 films against wavelength at different bath pH.

Extinction coefficient is defined as being the measurement of how strongly a substance absorbs light at a given wavelength. As seen in Figure 6, at a wavelength of ~ 400 nm, Al_2GaSe_3 thin films has extinction coefficient values of ~ 0.20 , 0.26 and 0.29 at pH values of 2.00, 3.00 and 4.00 respectively. The result showed that the highest light absorption takes place at pH of 4.00. Figure 6 further illustrates reduction in extinction coefficient of the thin films with wavelength increase.

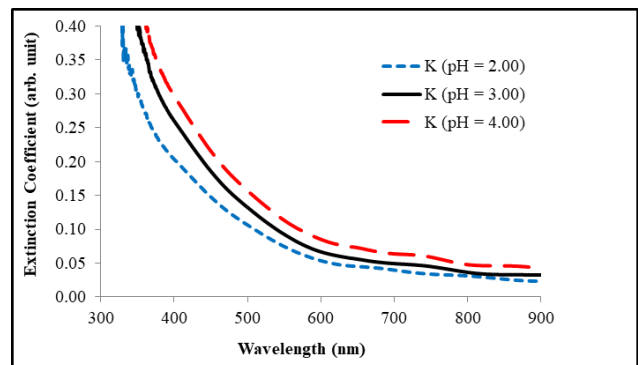


Figure 6: Plot of extinction coefficient versus wavelength for ED- Al_2GaSe_3 films at different bath pH.

Optical bandgaps determination of electrodeposited Al_2Se_3 , GaSe and Al_2GaSe_3 layers

Three compound semiconductors namely: Al_2Se_3 , GaSe and Al_2GaSe_3 were grown under similar growth conditions. Al_2Se_3 is a wider bandgap semiconductor with the bulk material having an energy bandgap of 3.10 eV (Balitskii *et al.*, 2013). As seen in the experimental investigations carried out in this work, the electrodeposited Al_2Se_3 thin films grown at a cathodic potential of 1200 mV and pH of 3.00 has an energy bandgap of 3.10 eV which corresponds to that of the bulk material. However, as seen in Figure 7, it was observed that despite the wider bandgap possessed by the material which makes it useful as a window layer in solar cells application, the Al_2Se_3 materials contained inherent defects which can limit its maximum output when used as a window material in solar cells applications.

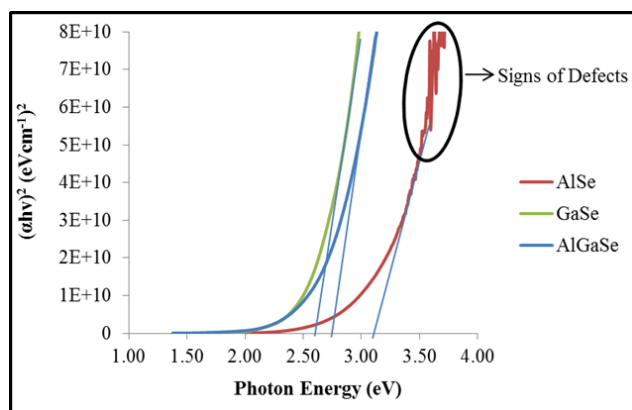


Figure 7: Energy band gaps estimation of ED - Al_2Se_3 , GaSe and Al_2GaSe_3 compound semiconductors

On the other hand, GaSe has a lesser bandgap of 2.60 eV when compared to Al_2Se_3 with a wider energy gap. However, the intrinsic defects observed in Al_2Se_3 as seen in Figure 7 were not present in GaSe. This shows that GaSe will be better in cell performance when compared to Al_2Se_3 . However, the lesser energy bandgap of GaSe when compared to Al_2Se_3 makes it less desirable as window layer. Several researches have experimentally demonstrated that the incorporation of Ga into compound semiconductors or as means of surface treatment of a compound semiconductor has the ability to passivate defects in the material (Ojo *et al.*, 2017; Olusola *et al.*, 2017a). With the ability of Ga to remove surface defects in a material, it thus becomes necessary to examine what happens when Ga salt is incorporated into the bulk of the material as a precursor for thin films growth. To improve the energy bandgap of GaSe and to make it further suitable as a window layer, there is therefore the need to incorporate Al salts into the GaSe electrolyte so as to form Al_2GaSe_3 electrolyte. With the incorporation of the Al precursor into the GaSe electrolytic bath, there was an improvement in the energy bandgap and removal of intrinsic defects in the newly formed Al_2GaSe_3 compound.

Researchers have successfully demonstrated several ways that can lead to the formation of ternary compound semiconductors. For instance, ZnCdS can be produced by using a single electrolytic bath containing Cd salts, Zn salts and S salts. Also, the production of ZnCdS can be

obtained from the intermixing of ZnS and CdS compound semiconductors when used as hetero – partners in junction formation (Olusola *et al.*, 2017b). It has been reported that the energy bandgap of CdZnS normally lies in – between that of CdS and ZnS (Hussein *et al.*, 2022). The same principle is also applicable to Al_2GaSe_3 . When an interface is created between Al_2Se_3 and GaSe, an intermix layer at the interface known as Al_2GaSe_3 is bound to be formed. The energy gap of the Al_2GaSe_3 intermix layer is therefore expected to lie in – between that of Al_2Se_3 and GaSe. This was what happened experimentally as seen in Figure 7. The energy bandgap of Al_2GaSe_3 (2.75 eV) lies in – between that of GaSe (2.60 eV) and Al_2Se_3 (3.10 eV). This result is an indication that Al_2GaSe_3 layers have been successfully grown.

Effect of pH variation on the electrical conductivity type and resistance of ED- Al_2GaSe_3

The sign of the PEC signals shows that Al_2GaSe_3 has p-type electrical conductivity. As shown in Figure 8, the magnitude increased from the pH of 2.00 to the pH of 3.00 and further reduces at the pH of 4.00. This indicates that pH of 3.00 where the magnitude of PEC cell signal is highest is a suitable pH for the growth of Al_2GaSe_3 layers.

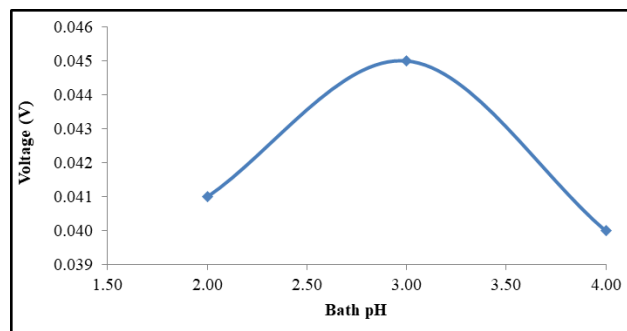


Figure 8: PEC cell signals of glass/FTO/ Al_2GaSe_3 thin film layers at different pH values.

The resistances of the thin films were determined using the current – voltage plot shown in Figure 9. The estimated resistances at pH of 2.00, 3.00 and 4.00 are $\sim 449 \Omega$, 272Ω , and 309Ω respectively. The results indicated that Al_2GaSe_3 grown at a pH of 3.00 possesses the least resistance as described in Figure 10.

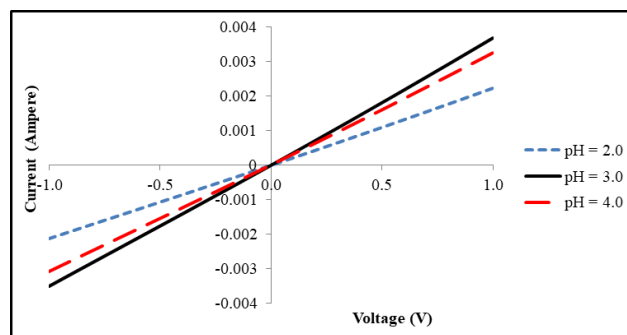


Figure 9: Typical current – voltage plot of Al_2GaSe_3 / electrolyte interface at varying pH values. (Note: Sodium thiosulphate $\{\text{Na}_2\text{S}_2\text{O}_3\}$ abbreviated as STS was used as the electrolyte to form the interface)

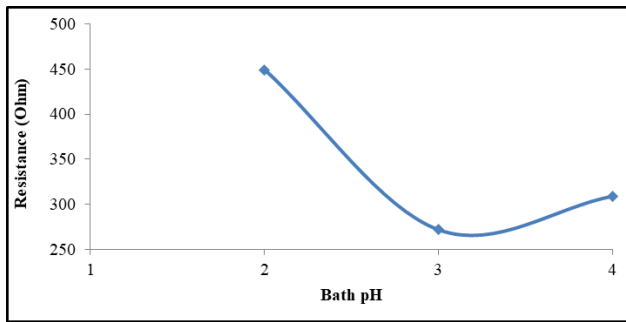


Figure 10: Typical plot showing the relationship between thin film resistance and pH values for ED- Al_2GaSe_3 films.

Influence of different measurement electrolytes on the current – voltage (I – V) characteristics of Al_2GaSe_3 films electrodeposited at varying bath pH

To investigate the effect of different measurement electrolytes on the thin film resistance, two different electrolytes namely: deionised water (abbreviated as DIW) and aqueous solution of sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) (abbreviated as STS) were used in the PEC cell measurement set – up. 2 g of $\text{Na}_2\text{S}_2\text{O}_3$ was dissolved in 100 mL of deionised water to form the aqueous $\text{Na}_2\text{S}_2\text{O}_3$ PEC electrolyte. The semiconductor was immersed in each of the electrolytes to form a solid liquid junction. With the aid of Iso – Tech current – voltage (I – V) analyser, the resistances of the thin films were determined using the current – voltage plots shown in Figures 11 – 13.

Figure 11 shows the current – voltage characteristics obtained when Al_2GaSe_3 layers grown at a pH of 2.0 were immersed in the two different electrolytes. From the I – V plot illustrated in Figure 11, resistance values of $\sim 449 \Omega$ and $\sim 1190 \Omega$ were obtained for Al_2GaSe_3 films immersed in electrolytes of $\text{Na}_2\text{S}_2\text{O}_3$ aqueous solution and deionised water respectively. Figure 12 shows the current – voltage characteristics obtained when Al_2GaSe_3 thin films deposited at a pH of 3.0 were immersed in the aqueous electrolytes. Resistance values of $\sim 272 \Omega$ and $\sim 442 \Omega$ were obtained from the I – V plot of Al_2GaSe_3 films immersed in electrolytes of $\text{Na}_2\text{S}_2\text{O}_3$ aqueous solution and deionised water respectively. For the Al_2GaSe_3 films deposited at a pH of 4.00, resistance values of $\sim 309 \Omega$ and $\sim 448 \Omega$ were obtained from the I – V plot of the films when immersed in electrolytes of $\text{Na}_2\text{S}_2\text{O}_3$ aqueous solution and deionised water respectively; this is illustrated in Figure 13.

Our experimental investigations revealed that the obtained resistances of the thin films are dependent on the electrolyte making the interface. Generally, the resistance obtained for films immersed in electrolyte from only deionised water has higher resistance than the ones obtained for films immersed in $\text{Na}_2\text{S}_2\text{O}_3$ electrolyte; this is diagrammatically illustrated in Figure 14. The reason for this could be as a result of the presence of Na ions in the bath used for PEC cell measurement. Na being a monovalent atom could further act as an unsolicited p – type dopants to the already existing p – type Al_2GaSe_3 layers to promote its conductivity enhancement, hence leading to a reduction in the material resistance. Considering the resistance values of Al_2GaSe_3 films deposited at different bath pH, it was

observed that the films deposited at pH of 3.00 have the least resistance under the different measurement conditions. Semiconductors with lesser resistance values are usually preferred for opto – electronic applications for efficiency enhancement. The least resistance and highest PEC signal magnitudes obtained at pH of 3.00 points to the fact that pH of 3.00 is an appropriate pH to grow a stoichiometric Al_2GaSe_3 material.

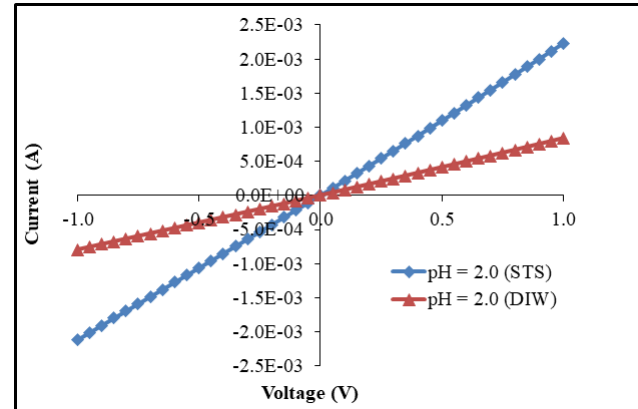


Figure 11: Typical current – voltage plot of Al_2GaSe_3 thin films grown at pH of 2.00 / electrolyte interface.

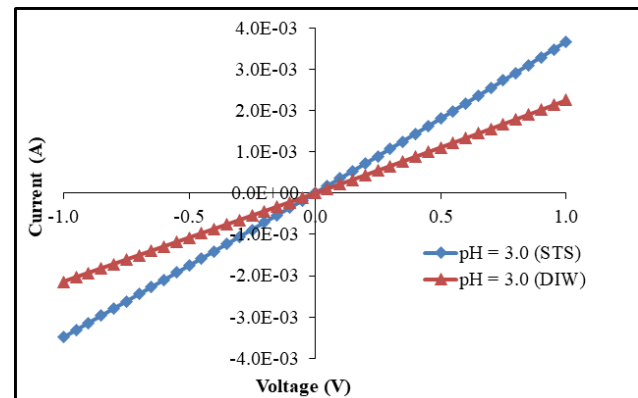


Figure 12: Typical current – voltage plot of Al_2GaSe_3 thin films grown at pH of 3.00 / electrolyte interface.

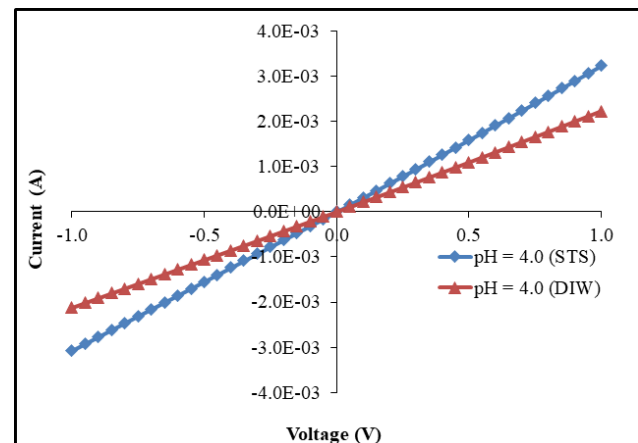


Figure 13: Typical current – voltage plot of Al_2GaSe_3 thin films grown at pH of 4.00 / electrolyte interface.

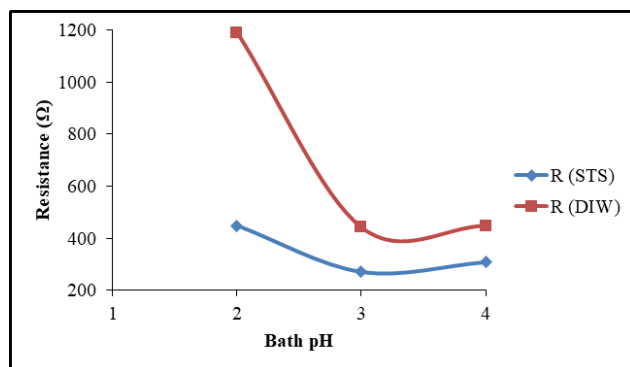


Figure 14: Typical plots showing the relationship between thin film resistance and pH values under different electrolyte measurement conditions.

CONCLUSIONS

Aluminum gallium selenide (Al_2GaSe_3) thin films were successfully grown through electrodeposition technique using a two-electrode system. The work has clearly revealed that the variation of the pH of an electrolytic bath to grow the materials has an effect on the visual appearance and opto – electronic properties of same material. It was discovered that at the lowest pH explored in this work, the material has a high band gap and the conductivity types achieved for all films grown within the explored pH range are positive. The increase in transmittance of the thin films with wavelength increase denotes that the Al_2GaSe_3 compound semiconductors will be good window materials for solar cells when other growth parameters such as deposition duration are properly optimized. The highest PEC cell signal and lowest resistance values obtained at bath pH of 3.00 revealed the possibility of getting a stoichiometric Al_2GaSe_3 layers at this pH. Future research work is aimed at optimizing the deposition potentials for the growth of Al_2GaSe_3 using the optimized pH and using relevant characterization techniques to determine the material properties.

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DECLARATION OF CONFLICT OF INTEREST

The authors have declared that no competing interests exist.

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