



Research Article

Optical and Structural Properties of TiO_2 and CuAlSe_2 Doped with Carbon Based Graphene for Photovoltaic Devices

Ladan Haruna Aminu^{1,*}, Ada Buba^{2,3}

¹Department of Physics, Saadu Zungur University, Bauchi, Nigeria

²CEMS Group, Department of Physics, Bauchi, Nigeria

³Department of Physics, University of Abuja, Abuja, Nigeria

Abstract

The preparation and characterization of copper aluminum diselenide (CuAlSe_2) and Titanium dioxide (TiO_2)-doped with carbon based graphene nanocomposite thin films were examined. The temperature, deposition time, and pH of the medium was varied by spin coating method. Samples were characterized by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and energy dispersive x-ray (EDX) and UV visible spectroscopy techniques for the structural, morphological, compositional features and photo-response for photovoltaic devices. The optical property revealed that CuAlSe_2 films have energy band gaps range 2.22-5.80 eV and 2.10-2.14 eV at room and peak temperature. The XRD patterns of CuAlSe_2 thin films showed peaks (101), (102), (006), (110), (108), and (116) corresponding to the formation of hexagonal phase of CuInSe_2 and the particle size $D \approx 38.20$ nm. XRD pattern of C: CuAlSe_2 and TiO_2 flakes were perfectly crystallized and the inter-planar spacing of ~ 0.053 nm. FESEM analysis indicated smooth and uniform structures. The graphene- TiO_2 displayed glistening surfaces due to less density of electronic trap states and improved absorption in the UV region. The resistivity of the samples were 1.95s/m and 12.93s/m for GO and GO- TiO_2 . The results indicated that the average electron mobility depends on the probability of the electrons in the conduction band and as the quasi-Fermi level approaches the CB, a higher current, power supply and lower resistivity level.

Keywords

Graphite, Graphene, CuAlSe_2 C, TiO_2 -GO, CuSe, Spin Coating

1. Introduction

Demand for sustainable electrical power and energy for domestic and industrial activities are always on the rise. The conventional fossil based energy systems have their disadvantages especially environmental degradation. TiO_2 and

CuSe based solar cells are cost effective and are not highly efficient especially in energy conversions. The three crystal-line forms of TiO_2 are anatase, rutile, and brookite. The 2p oxygen and 3d titanium hybridized to form the valence band

*Correspondence: Ladan Haruna Aminu (Ladansons@gmail.com), Ladan Haruna Aminu (Ladan@sazu.edu.ng)

Received: 11 June 2025; Accepted: 4 February 2026; Published: 27 February 2026



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of the TiO₂. CuSe exists in cubic, orthorhombic, tetragonal or monoclinic forms depending on the stoichiometry (with α -Cu₂Se, CuSe, Cu₃Se₂ and Cu₂Se and non-stoichiometric Cu_{2-x}Se) and the growth techniques with change of physical and chemical properties. CuSe is used as super ionic conductor, electro-optical filter, thermo electric converters, lasers, photovoltaic and electrochemical cell. CuAlSe₂ superior electrical and optical properties are desirable for optical applications. Hexagonal graphene oxide derived from graphite is sp² allotrope of carbon. It has a high tensile strength, high charge mobility, conductivity, transparency, and flexibility for diodes and optical devices [1-5].

The silicon based devices are approaching their theoretical upper limit, have limited operational life-time, high cost and production technique, poor transmission of photons and thermalization of hot carriers. Tell and Wiegand first studied the photo-detection properties of Cu₂Se-AgInSe₂ heterojunctions diodes formed by vacuum evaporation of p-type Cu₂Se on n-type AgInSe₂ substrates. The applications of semiconducting metallic oxides (SMOs) such as TiO₂, ZnO, NiO, SnO₂, SrTiO₃, Nb₂O₅, ZrO₂ and CuO as carrier transporters enhance the efficiency due to reduction in density of electronic trap states, improves carrier mobility, and conductivity as transporters in regular-structure Perovskite solar cells (PSCs) and CuSe. Modifying the surface features have proved effective in enhancing the key electron-transport properties and performance. GO as optimizer reduces density of electronic trap states, instability under UV light, hysteresis, safety and cost [5, 6].

Studies on improving the performance and efficiency of solar energy as a viable option has been on the rise. There is need for manufacturing high quality carbon based CuAlSe₂ and TiO₂ thin films for energy conversion. In this study, synthesis, producing GO-TiO₂, CuSe and characterizing thin films for energy conversion is investigated using X-ray diffraction (XRD), field emission scanning electron microscopy (-FESEM), energy dispersive x-ray (EDX), UV visible spectroscopy and FTIR analysis for enhanced industrial and research activities.

2. Experimental Details

2.1. Materials

Natural graphite powder, TiO₂ (Solaronix, Switzerland), potassium permanganate (KMnO₄), Sodium nitrate (NaNO₃), Sulphuric acid H₂SO₄ (98%), purified water, Hydrogen peroxide (H₂O₂, 30%) and Hydrochloric acid (HCl), Aluminum tri-sulphate (Al₂(SO₄)₃·14H₂O), Copper chloride (CuCl₂·2H₂O), Sodium Selenosulphate (Na₂SeSO₄), disodium ethylene diaminetetraacetic acid [Na₂EDTA] (C₁₀H₁₄N₂Na₂O₈·H₂O) and Ammonia (NH₃) Solution. All the analytical grades were used without further purification.

2.2. Synthesis of Reduced Graphene Oxide

Synthesis and characterization of graphene oxide and reduced graphene oxide thin films were described elsewhere [7].

In brief, 2g of natural graphite, 10ml of conc. H₂SO₄, and 1g of KMnO₄ were mixed together and stirred with 1.5g of NaNO₃ in a 500ml beaker. At the temperature below 5°C, the mixture was stirred using a magnetic stirrer for half an hour. 100 ml of purified water was added gradually to form a dark brown color, with increasing temperature to 45°C. The mixture was kept on a magnetic stirrer at 95 °C and continuous stirring was done until a bright-yellow mixture was obtained. 10 ml H₂O₂ (30%) was added to the solution to form a golden yellow colouration. After centrifugation at 1000 rpm for 5 minutes and separation, the purification process to remove metal ions/acid radicals were carried out using 10% HCl at pH ~7. Sample was heated thermal method to have reduced graphene oxide (RGO).

2.3. Preparing TiO₂, RGO and CuAlSe₂ Composite Thin Films

Procedure for the preparation and characterization of CuInSe₂ thin films were explained in detail by Garg et al. in 1988. In brief, the fluorine-doped tin oxide (FTO Solaronix) 7 Ω/sq glass substrates were ultrasonically cleaned in acetone and deionized water. The TiO₂ nanoparticles (Solaronix) and RGO were mixed in the ratio 10:1. Spin coating method was carried out at 1000 rpm for 30s using a spin coater. The films were annealed at 300 °C, 500 °C and 600 °C. The same procedures were used to prepare CuAlSe₂ thin films. Aluminum tri-sulphate, Copper chloride and sodium selenosulphate were sources of Al, Cu and Se ions respectively. 3ml of CuCl₂·2H₂O and Al₂(SO₄)₃·14H₂O (0.1M) solutions were complexed with 5ml of Na₂EDTA (0.1M), TiO₂ and RGO. The absorbance, reflectance and transmittance with wavelength were studied at 50 °C, 60 and 70 °C respectively. The energy band gap, optical conductivities and refractive index against photon energy were determined for samples RGO (A), GO (B), RGO-TiO₂ (C), and GO-TiO₂ (D) [5] (Figures 1, 2).

2.4. Characterization of the Samples

The SEM revealed the morphological characteristics and the elemental compositions using SEM EVO LS 10-electron microscopy (10-100x, 20 kV, 10mA). The phase composition was identified using XRD (ARL XTRA thermo fisher) with CuK_α radiation ($\lambda = 0.154$ nm) at 45kV and 40 mA with a diffraction angle between 0 ° and 90 ° to show the crystalline phases. The crystallite size was determined from the broadenings of the X-ray spectral peaks by using Debye Scherrer's expression. UV visible spectroscopy analysis (T80+) techniques performed for the photo-response.

Electrical characterization of the thin films were performed using four point probe (4200 Keithley) at room temperature.

The resistivity ρ of the graphite thin films were determined by

$$\rho = R_s t$$

$$R_s = \frac{\rho}{t} = \frac{\pi}{\ln 2} \left(\frac{V}{I} \right) = K \left(\frac{V}{I} \right) = \left(\frac{V}{I} \right) 4.532$$

Where V is the measured voltage, Where R_s is the sheet resistance and t is the films thickness, I is the current and K is the geometric factor which depends upon the configuration of the probes ($K = 4.532$).



Figure 1. Samples and the thin films Carbon (A), CuAlSe₂ (B), Coal (C) and TiO₂ doped with RGO (D) TiO₂ on FTO glass substrate (solaronix).



Figure 2. Starting materials and the thin films carbon, TiO₂ on FTO glass substrate.

3. Results and Discussion

3.1. XRD Analysis of TiO₂-RGO and CuAlSe₂-RGO Thin Films

XRD is used to determine the crystal structures, phases and impurity in substances by irradiating the sample. XRD pattern

showed that RGO-CuAlSe₂, and RGO-TiO₂ flakes were perfectly crystallized and closely packed. It showed a sharp diffraction peak at $2\theta = 27^\circ$ and at reflection plane (002) and the formation of nano-crystalline Cu_{2-x}Se particles of the size range from 10 nm through 100-150 nm [16, 17].

The XRD patterns of CuSe thin films showed peaks (101), (102), (006), (110), (108), and (116). These peaks were compared with the JCPDS diffraction patterns to JCPDS Data (00-020-1020). The peaks corresponds to the formation of hexagonal phase of CuInSe₂ (Figures 3-5). The bond lengths in bulk CuInSe₂ are 2.45 Å (Cu-Se bond) and 2.61 Å (In-Se bond) [2, 4, 16, 18]. The particle size (D) can be determined, using the Scherrer formula, if the wavelength (λ), full width at half maximum (FWHM) of the peaks (β), and the diffracting angle (θ) is known.

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

Where D is 38.2 nm similar to reported values 37.5 nm and 38.0 nm [17, 19].

For the RGO, XRD diffractogram indicated the crystal structure at peak 130 for RGO. When graphite is oxidized to graphene oxide due to the intercalated epoxy, hydroxyl, carbonyl and carboxyl functional groups. The XRD peak shift from about 130 to ~ 450 and no sharp peak observed from 100 to ~ 270 resulting from incomplete graphite oxidation (Figure 3a and b). The compositional analysis of coal used showed graphite, Quartz, Muscovite and Chlorite at 15%, 11%, 10% and 5% respectively.

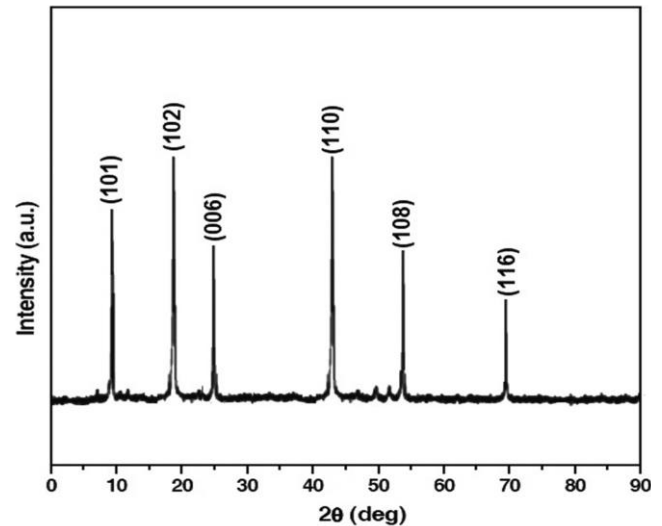


Figure 3. X-ray diffraction pattern of CuInSe₂.

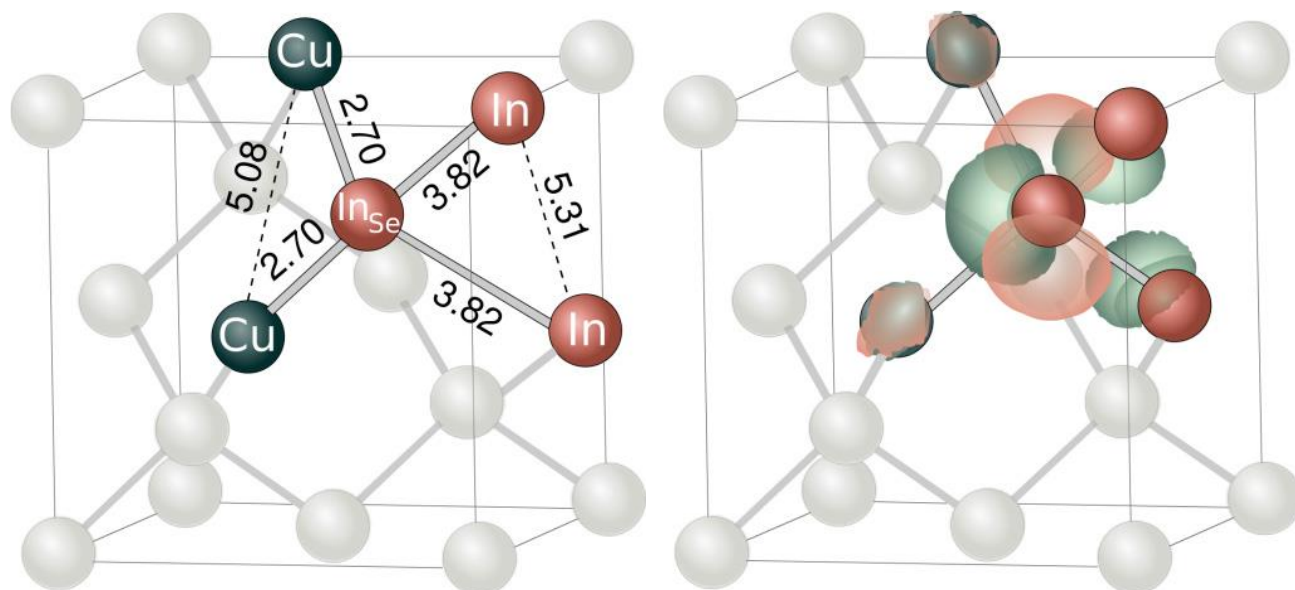


Figure 4. The interatomic distances in A^0 around defect atom in CuInSe_2 [18].

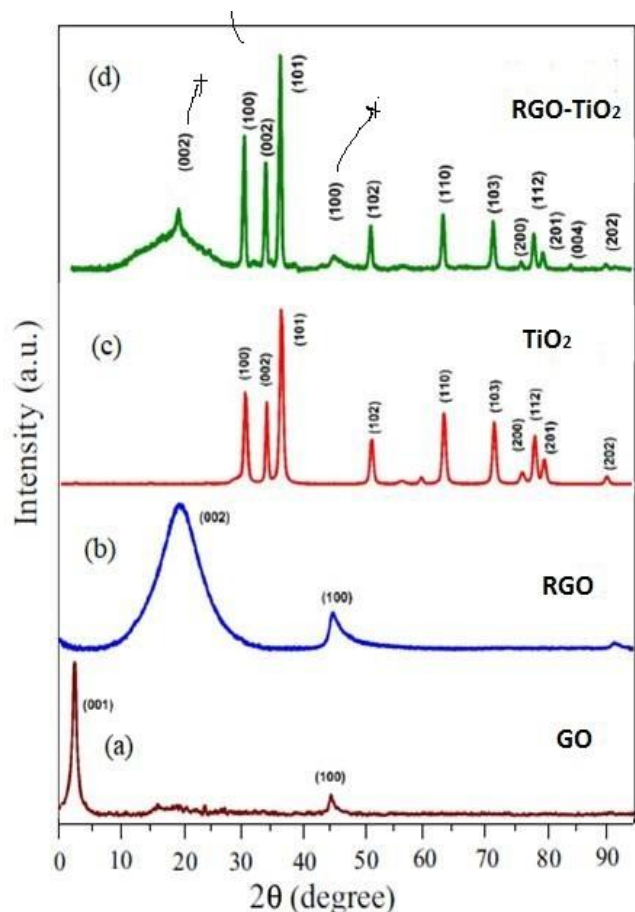


Figure 5. XRD Analysis of the RGO-TiO_2 , RGO , GO on quartz substrate.

3.2. SEM of the TiO_2 and CuAlSe_2 - RGO Thin Films

SEM showed the surface morphology and micro structural

features of the samples at 250 and 1000x (Figures 6, 7). It is reflective when exposed to light rays, a flat-densely packed spongy structures in layers. An indication of multiple nucleation due to reactivity of the carbon and the elements. They displayed a glistening surfaces can be attributed to the SMO-TiO_2 and CuAlSe_2 with high density of electronic trap states. The surface image of the particles are uniformly distributed showing the granular nature of the nanoparticles without selenium-related defects. An indication of the spongy and rough surface an interesting case among the selenium-related defects is the selenium interstitial in the dumbbell configuration [4, 7, 18, 19] the same situation is reflected in the characterization of p- CuInSe_2 films for photovoltaic grown by a chemical deposition technique [20].

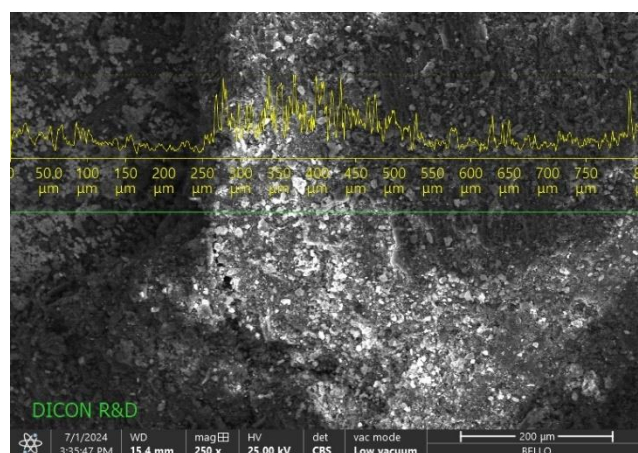


Figure 6. SEM micrograph of samples $\text{RGO-CuAlSe}_2\text{-TiO}_2$ at magnification of 250x.

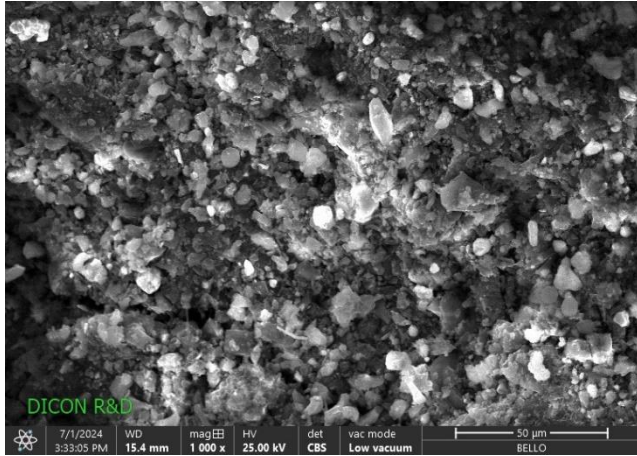


Figure 7. SEM micrograph of samples RGO-CuAlSe₂-TiO₂ at magnification of 1000x.

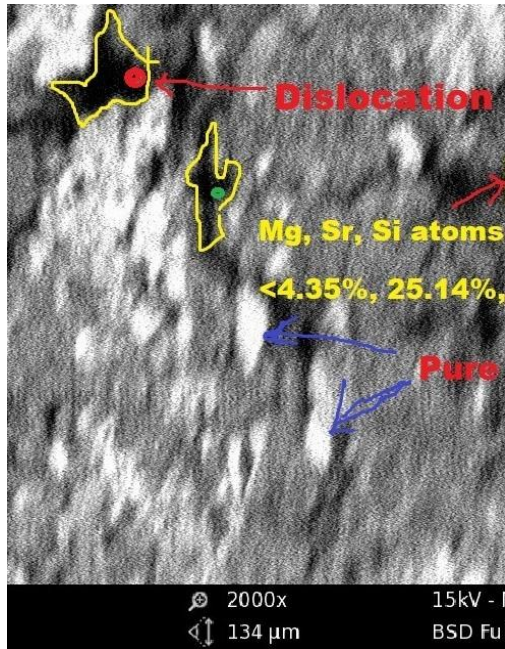


Figure 8. (a) Crystal defects in RGO-CuAlSe₂-TiO₂.

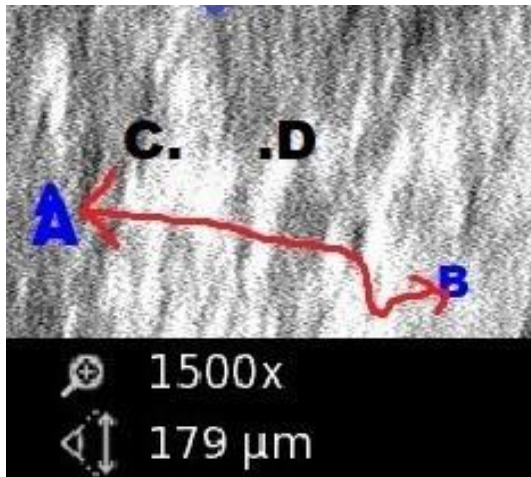


Figure 9. Continuity test and electrical continuity point A to B.

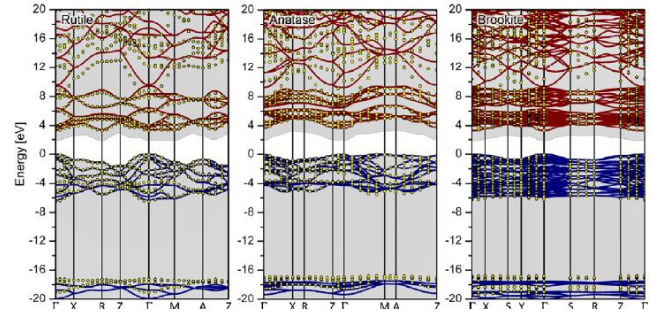


Figure 10. TiO₂ band-structures: Rutile, Anatase and-brookite [9].

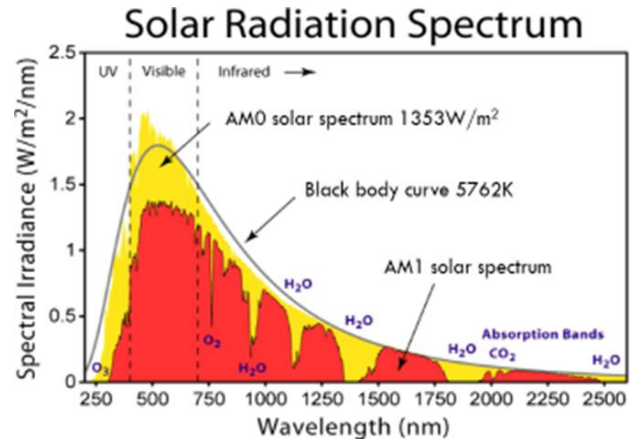


Figure 11. Solar Energy Spectrum [8].

3.3. The Conductivity of the TiO₂-GO and CuAlSe₂-GO Thin Films

The AC electrical conductivity, indicated that the conduction depends both on the frequency and the temperature. It implies that the temperature dependent conductivity confirmed the semiconducting characteristics of the films [17]. The electron transport in RGO-TiO₂ is governed by detrapping from sub-band gap states deep in the tail of the density-of-states to conduction band (CB) in line with multi-trapping mechanism. Figure 9 showed the continuity test and electrical continuity point, A to B. Trap states play crucial role in the occupation of the sub-band gap states at a particular energy EA which can be expressed by the Fermi-Dirac distribution function:

$$F(E_A - E_{Fn}) = \frac{1}{1 + e^{(E_A - E_{Fn})/K_b T}}$$

The density of carriers at this energy can be expressed as, $n_A = N_A E_A$. Assuming that electrons can only be transported via the CB, then density of electrons in the CB (n_{CB}) varies with the position of the quasi-Fermi level for the electrons (E_{Fn}) the conductivity of the film is proportional to n_{CB} it is given by:

$$n_{CB} = N_{CB} e^{(E_A - E_{Fn})/K_b T}$$

The conductivity can also be expressed in terms of the Fermi energy, E_F as

$$\sigma(E_F) = e\mu_e n_e(E_F)$$

Where NA is the total number of available sites at this energy, $f(E)$ is the probability that a state of energy E is occupied, E_F is the Fermi energy, K_b is the Boltzmann's constant, T is absolute temperature, e is the electronic charge, μ_e represent the electron mobility, and n_e is the electron carrier density.

The average electron mobility depends upon the probability of the electrons being in the CB, and conductivity increases as the quasi-Fermi level for electrons approaches the CB energy. It implies that any modification and doping that eliminates deep trap states will increase the conductivity of the samples and higher current output from the device will be achieved when applied in optical and photovoltaic cells. The application of RGO-CuAlSe₂-TiO₂ thin films in optoelectronic devices, solar cells and transparent electrodes is governed by the sheet resistance and visible-light transmission. Each of these criteria having its own unique requirements in improving the optoelectronic properties. Either of these properties can be fine-tuned to the desired value by changing the thickness of the graphene film. The smaller the sheet resistance, the higher the transmission of light through it [9-12] and improve photo detection properties of Cu₂Se - AgInSe₂ heterojunctions [21].

RGO reduces internal light scattering in TiO₂ and CuAlSe₂ based devices and improve optical response by shifting absorption to the visible region of the electromagnetic spectrum at wavelength between 500nm and 750nm, AM 0 solar spectrum 1353W/m² [8].

Optical response of the TiO₂ and CuAlSe₂ thin films from 300 to 700 nm at deposition temperature of 60 °C, 70 °C and 80 °C increases with temperature indicating that these materials can be used in the energy conversion system, optoelectronics and solar cell applications. CuAlSe₂-RGO; and TiO₂-RGO band gap decreases with temperature increase. It optimizes the value in the range 2.10 to 2.15 eV. It has different crystalline structures rutile, anatase and brookite (Figure 10) [9].

The presence of defects (Figure 8) influence the electrical conduction and upon thermal reduction to RGO, the conductivity improved. The electrical characterization of the thin films were performed using four point probe (4200 Keithley) at room temperature showed that electrical conductivity depends on chemical composition, purity, crystal structure and temperature dependent currents [13, 14].

Figures 12 and 13 showed graph of film thickness versus the deposition temperature and Absorption coefficient squared $\times 10^{12}$ (/m²) versus photon energy for C at 600C of the radiation at increasing order of absorbance of sample A, B and C having the deposition temperature of 80 °C, 70 °C, and 60 °C. The highest absorbance of visible radiation occurred at 60 °C.

Figures 14 and 15 indicates the transmittance and reflectance versus wavelength. Only sample A has the lowest and B has highest transmittance in the UV region. The reflectance at

360nm and 460nm is high for A, B and C which increases temperature and C being the highest. Figures 16 and 17 represents the absorbance against wavelengths of A, B and C and is the optical conductivity against photo energy of the sample which increases linearly with the photon energy.

The principal optical properties presented in the visible and near-IR ranges showed direct transitions in the range of 2.10-2.36 eV in line (Table 1) with similar results and graphene for transparent electrodes and organic electronic devices [15, 16, 19]. Sample A (60 °C) has the highest conductivity while sample C (80 °C) has lowest. The narrower the optical band gap the better in trapping solar energy rays for photo conversion process. Figure 18 is the refractive index n of the samples versus photon energy which indicated similar pattern at varying energy.

Optical characterization of CuIn_{1-x}GaxSe₂ alloy and polycrystalline thin films are promising materials for photovoltaic applications due to their higher conductivity and optical properties [22]. Sample C has the highest value. The thin films can be used in coating glasses since they have high n values.

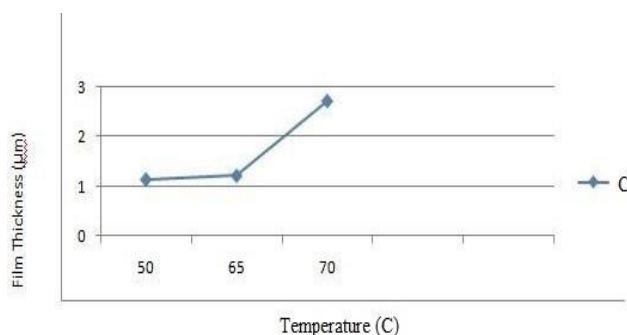


Figure 12. A graph of film thickness versus the deposition temperature.

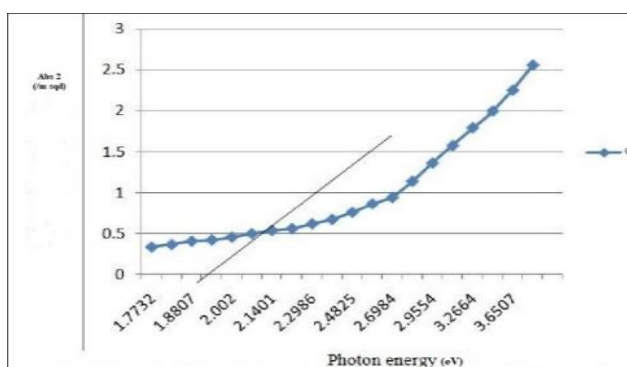


Figure 13. Absorption coefficient squared $\times 10^{12}$ (/m²) versus photon energy for C at 60°C.

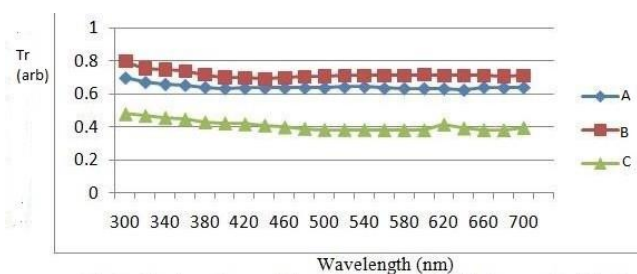


Figure 14. The transmittance versus wavelength (nm) of the radiation.

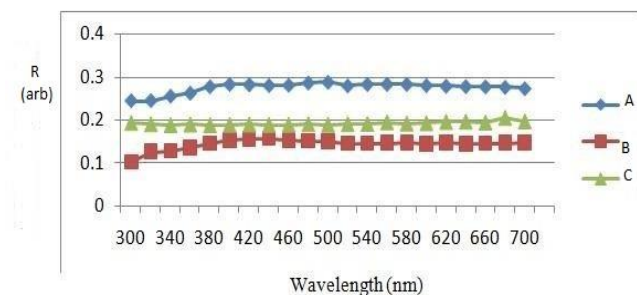


Figure 15. The reflectance versus wavelength (nm) of the radiation.

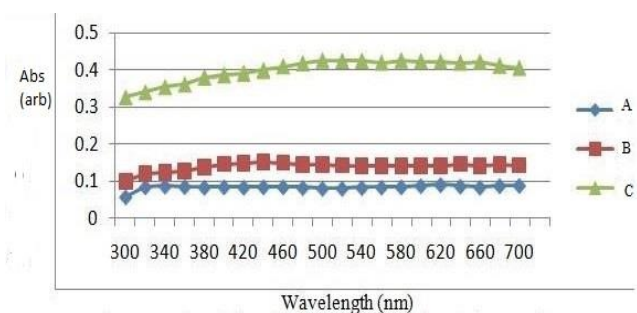


Figure 16. Absorbance versus wavelengths sample A, B and C.

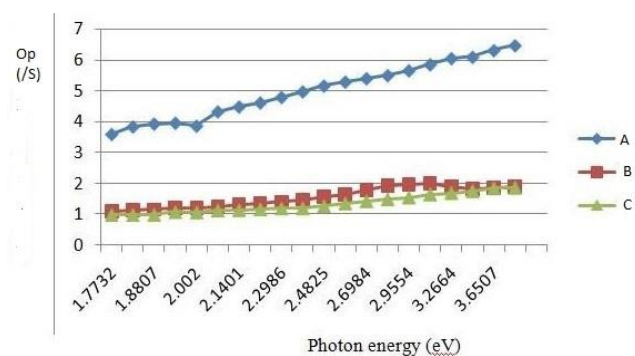


Figure 17. Optical conductivity against photo energy of samples A, B and C.

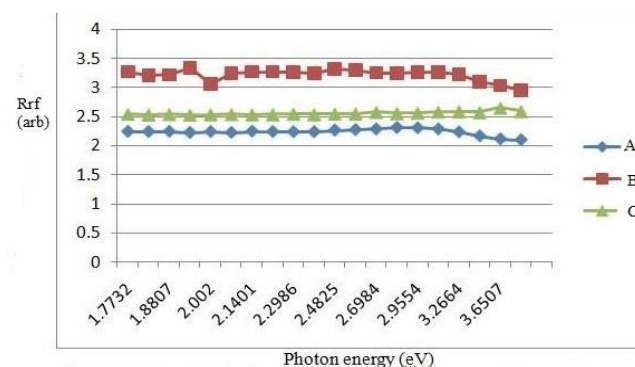


Figure 18. Refractive index of the samples against the photon energy.

Table 1. The conductivity of the GO and RGO thin films.

Graphene	Thickness t (μm)	V (mv)	I (A) $\times 10^{-9}$	R_s (Ω/sq) $\times 10^6$	ρ (Ωm)	$\frac{1}{\rho}$ S/m	References
TiO ₂	0.022	28.0	5.47	23.20	0.5104	1.93	This study
RGO-CuAlSe ₂	0.015	50.0	44.01	5.15	0.0773	12.94	This study
RGO-TiO ₂	0.022	14.6	3.70	17.88	0.3934	2.54	This study
CuAlSe ₂	0.015	53.0	90.65	2.65	0.0398	25.13	This study
GO-NGF	0.027	25.0	4.95	22.90	0.6183	1.62	[7]
RGO-NGF	50.0	45.69	4.95	0.1330	7.52		
RGO	0.025	-	-	0.0179	-	23.30	[23]
CuSe	-	-	-	-	10.0	0.001	[19]
CuInSe ₂	-	300.0	-	-	1.03	0.97	[20]

4. Conclusion

In this study, synthesis and characterization of RGO by modified Hummers method to produce CuAlSe₂ and TiO₂ thin films and annealed at different temperature. The samples were characterized for the structural, morphological, compositional features and photo response for photovoltaic devices. The optical property revealed that CuAlSe₂ films have energy band gaps range 2.22-5.80 and 2.10-2.14 eV at room and peak temperature. XRD pattern of C: CuAlSe₂ and TiO₂ flakes were perfectly crystallized. FESEM analysis indicated smooth, and uniform structures. The RGO-TiO₂ displayed glistening surfaces due to less density of electronic trap states and improved absorption in the UV region. The resistivity of the samples were 1.96, 12.94, 2.54 and 25.13 s/m for GO, RGO, GO-TiO₂ and RGO-TiO₂ respectively.

The optical properties showed that the films are useful in PVCs applications, thermal window coatings and optoelectronic devices. RGO reduces internal light scattering in TiO₂ and CuAlSe₂ based devices, and improve the optical response by shifting the absorption to the visible region of the electromagnetic spectrum these materials can be used in the energy conversion system, optoelectronics and solar cell applications. CuAlSe₂-GO; and TiO₂-GO band gap energy decreases as the temperature increases, a direct band gap and RGO optimizes the value in the range 2.10 to 2.15 eV. The transmittance and reflectance versus wavelength. Only sample A has the lowest and B has highest transmittance in the UV region. The reflectance at 360nm and 460nm is high for A, B and C which increases temperature and C being the highest. The optical conductivity against photo energy of the sample which increases linearly with the photon energy. The narrower the optical band gap the better in trapping solar energy rays for photo conversion process samples with highest optical band gap energy will trap more energy from the sun. Therefore, it is expected to grow films below and at room temperature to enhance their conductivities. The refractive index *n* of the samples versus photon energy which indicated similar pattern at varying energy and can used in coating glasses.

The results indicated that the average electron mobility depends on the probability of the electrons in the conduction band (CB) and as the quasi-Fermi level approaches the CB, a higher current, power supply and lower resistivity level. Polycrystalline thin films are promising materials for photovoltaic applications (due to their higher conductivity and optical properties) and for academic and industrial research at a reduced cost.

Abbreviations

CuAlSe ₂	Copper Aluminum Diselenide
TiO ₂	Titanium Dioxide
XRD	X-ray Diffraction
FESEM	Field Emission Scanning Electron Microscopy

EDX	Energy Dispersive x-ray
SMOs	Semiconducting Metallic Oxides
PSCs	Perovskite solar cells
Al ₂ (SO ₄) ₃ .14H ₂ O	Aluminum Trisulphate
CuCl ₂ .2H ₂ O	Copper Chloride
Na ₂ SeSO ₄	Sodium Selenosulphate
[Na ₂ EDTA]	Disodium Ethylene Diaminetetraacetic Acid
(C ₁₀ H ₁₄ N ₂ Na ₂ O ₈ .H ₂ O)	Reduced Graphene Oxide
RGO	Fluorine-doped tin Oxide
FTO	Scanning Electron Microscopy
SEM	Full Width at Half Maximum
FWHM	Density of Electrons
<i>n</i> _{CB}	Quasi-Fermi Level
<i>E</i> _{Fn}	Boltzmann's Constant
<i>K</i> _b	GO
Sample A	RGO
Sample B	CuAlSe ₂ -GO
Sample C	TiO ₂ -GO
Sample D	Conduction Band
CB	

Author Contributions

Ladan Haruna Aminu: Formal Analysis, Project administration, Resources, Software, Supervision, Validation, Visualization

Conflicts of Interest

There is no conflict of interest on the publication of this paper.

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