

## Analyzing the structural, optoelectronic, and optical properties of ZrS nanostructured material by adding silver (Ag) dopant to its lattice.

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### Abstract

In this study, a range of (0.01-0.03) mol was used to synthesize silver doped ZrS through electrodeposition. A three-electrode system was employed in the synthesis. Platinum is used for the anode, silver and silver chloride (Ag/AgCl) are used for the reference electrode, and FTO (fluorine-doped tin oxide) is used for the cathode. The ZrS and Ag/ZrS XRD patterns showed the materials are polycrystalline with distinct phase orientation planes. An intense peak, indexed at (111) and showing values between 23.59° and 62.62°, displays a heavily dampened peak plane at (111). The micrograph of the ZrS material shows a presence of hexagonal structure material along with precipitate. The presence of various silver concentrations results in particle clumping and a uniformly coated substrate surface. In the visible region, the Ag/ZrS material displays a distinct absorption band, which is attributed to the silver surface plasmon resonance. The wavelength of the surface plasmon resonance band is determined by the size, shape, and dielectric properties of the silver material and the surrounding ZrS material. The energy band structure of ZrS increases from 2.32 to 2.51 eV with increasing silver molarity due to silver incorporation within the ZrS lattice.

**Keyword:** X-ray diffraction patterns, optoelectronic, Energy dispersive X-ray, bandgap, Scanning electron microscopy, optical,

### Introduction

The primary global challenges arising from pollution and the greenhouse effect due to fossil fuel combustion rather than renewable energy utilization are energy and environmental issues (Alzoubi *et al.*, 2021; Elhalim *et al.*, 2021). To combat the surge in energy consumption resulting from population growth and developing nations' demands, we must replace non-renewable energy sources. Finding clean and

sustainable renewable energy sources is now an urgent priority (Alnehia *et al.*, 2023; Article, 2024; Bencherif *et al.*, 2022; Bouarissa *et al.*, 2021). Solar energy is considered by researchers as a cost-effective and renewable option with abundant availability and high output efficiencies. The solar photovoltaic industry relies heavily on the distribution and intensity of solar radiation. Solar thermal applications use solar energy as a heat or electricity source in technologies like concentrated fuel cells and

solar power plants (Hasan *et al.*, 2023; Ikhioya, 2024; Ikhioya and Nkele, 2024). All technologies harness sunlight and convert it into different forms. Solar energy can be converted into solar fuel through photosynthesis, as an example. Through photosynthesis, plants store solar energy by producing protons and electrons, which can be transformed into H<sub>2</sub> and CH<sub>4</sub>. Biomass photosynthesis only uses 11% of solar energy. Photovoltaics convert photons into electrons for electricity, while solar thermal applications absorb and convert photons into heat (Jimin Shang *et al.*, 2019; Kashif *et al.*, 2022s; Kumar *et al.*, 2016; Li *et al.*, 2019; Mattinen *et al.*, 2019). The heat is used to warm a working fluid, which can then be collected and used to directly heat spaces and water. However, the energy conversion may not be enough, so we need to improve production efficiency by developing fuel from water and carbon dioxide with biological-inspired nanoscale assemblies, innovative configurations of natural photosynthetic pathways, and genetic engineering to boost biomass production (Moustafa *et al.*, 2021, 2022; Nnanna *et al.*, 2024; Sharma *et al.*, 2024; Shetti *et al.*, 2019). Photovoltaic systems still face a significant challenge in aligning intermittent energy production with fluctuating power demand. To tackle this challenge, an option is to integrate a storage element into these sporadic energy sources.

In two dimensions, dichalcogenides are a versatile group of materials with diverse properties and numerous potential applications. TMDCs have gained popularity following the rise of graphene, the original 2D material made of carbon with semi-metallic properties. Scientists have conducted extensive research on group 6 elements such as molybdenum and tungsten, focusing on their semiconducting sulfides and selenides and their potential applications (Tian *et al.*, 2022; Tripathi *et al.*, 2021;

Valussi *et al.*, 2021; Ye *et al.*, 2023). Recently, attention has shifted to a group of semiconducting materials, namely HfSe<sub>2</sub>, ZrSe<sub>2</sub>, HfS<sub>2</sub>, and ZrS<sub>2</sub>, because of their potential in semiconductor applications. Despite limited studies, both ZrS<sub>2</sub> and HfS<sub>2</sub> are likely indirect band gap semiconductors, regardless of their thickness. The reported band gaps of 1.8–1.7 eV for ZrS<sub>2</sub> and 2.1–1.8 eV for HfS<sub>2</sub> in bulk are suitable for different semiconductor applications.

Tian *et al.* [20] successfully showed the deposition of high-quality, uniform ZrS<sub>2</sub> films on c-plane sapphire substrates using chemical vapor deposition. The atomic interface between ZrS<sub>2</sub> and sapphire exhibits an exceptional level of sharpness. ZrS<sub>2</sub> films show optoelectronic applications through photodetector devices. The ZrS<sub>2</sub> photodetectors show exceptional performance, with a light on/off ratio of 106 and a specific directivity of  $2.6 \times 10^{12}$  Jones, which is the highest among other group-IVB two-dimensional Transition-metal dichalcogenides (TMDs). Mattinen *et al.* (Mattinen *et al.*, 2019) introduced a method that enables the creation of uniform films on different substrates with precise control over thickness. To increase the process scale, industry-compatible precursors and temperatures around 400 °C can be used. The deposited ZrS<sub>2</sub> and HfS<sub>2</sub> films are smooth, crystalline, with oxygen as the primary impurity. Applying an AlxSiyOz layer in a vacuum environment eliminated ZrS<sub>2</sub> and HfS<sub>2</sub>'s sensitivity to oxidation and reduced impurities. ZrS<sub>2</sub> and HfS<sub>2</sub> photodetectors exhibit exceptional performance, remaining stable even in ambient conditions. The photo-responsively achieved is comparable to ZrS<sub>2</sub> and HfS<sub>2</sub> thin films or single flakes deposited at higher temperatures, but the response speed seems limited by photo-gating, as expected for 2D photo-detectors. The initial atomic layer deposition (ALD) techniques for

ZrS<sub>2</sub> and HfS<sub>2</sub> open up new possibilities for exploring their semiconductor applications.

ZrS doped with silver displays excellent carrier mobility and a large bandgap as a semiconductor. Extensive studies have been carried out to explore its potential applications in optoelectronics, photovoltaics, and sensing. Methods like chemical vapor deposition, molecular beam epitaxy, sol-gel processing, and electrochemical deposition technique (Ikhioya *et al.*, 2020; Ikechukwu & Ikhioya, 2024; Ikhioya, 2015b, 2015a; Ikhioya *et al.*, 2015, 2020, 2021; Ikhioya and Ekpunobi, 2014b, 2014a; Ikhioya and Nkele, 2023a, 2023b; Ikhioya *et al.*, 2023) can synthesize silver-doped ZrS. The doping level and distribution can be controlled by adjusting the synthesis parameters. The addition of silver to ZrS shows promise in photocatalysis, energy storage, and biomedicine. Scientists are investigating how it can be used in flexible electronics and transparent conductors (Alnehia *et al.*, 2023; Sharma *et al.*, 2024; Shetti *et al.*, 2019).

The primary aim of this research is to investigate a nanostructured material produced by incorporating silver (Ag) into zirconium sulfide (ZrS). This material exhibits specific attributes and has practical applications in diverse areas. Silver-doped ZrS nanostructures have shown significant potential in optoelectronics, photocatalysis, and energy storage. Their unique optical and electrical properties can be advantageous for solar cells, photodetectors, and batteries.

### Experimental Procedures

In this study, we employed the electrochemical deposition technique (ECD). The electrochemical bath system consists of ZrOCl<sub>2</sub>.8H<sub>2</sub>O (Zr<sup>2+</sup>) as the cation source, C<sub>2</sub>H<sub>5</sub>NS (S<sup>2-</sup>) as the anion source, and distilled water, all combined in a 100-mL beaker. A magnetic stirrer was used to stir the

reaction bath. The power supply generated the electric field (DC voltage), with the cathode made of conducting glass and the anode composed of carbon and fluorine electrodes. Uniform thin film deposition through electrochemical deposition has been achieved. The FTO-coated working electrode, measuring 2.5 cm × 1.5 cm, was fragmented and cleaned using dish washing liquid. To synthesize Ag/ZrS, measure 0.01-0.03 mol of AgNO<sub>3</sub> precursor, a 0.1 mol ZrOCl<sub>2</sub>.8H<sub>2</sub>O solution in a 100-mL beaker, and a 0.5 mol C<sub>2</sub>H<sub>5</sub>NS precursor. The synthesis employs a three-electrode system. The anode is made of platinum, the reference electrode is made of silver and silver chloride (Ag/AgCl), and the cathode is made of FTO (fluorine-doped tin oxide). The FTO-coated substrate housed the counter and reference electrodes vertically in the beaker. The synthesis involved maintaining a potentiostatic condition of -200 mV versus SCE for 5 seconds. The synthesized films were cleaned and dried using a hand dryer. Target materials were poured into beakers alongside equal amounts of precursors during the synthesis process. The films underwent a 20-minute annealing process to remove internal stress. The optical, structural, elemental analysis, and electrical properties of the deposited materials were thoroughly examined using appropriate tools.

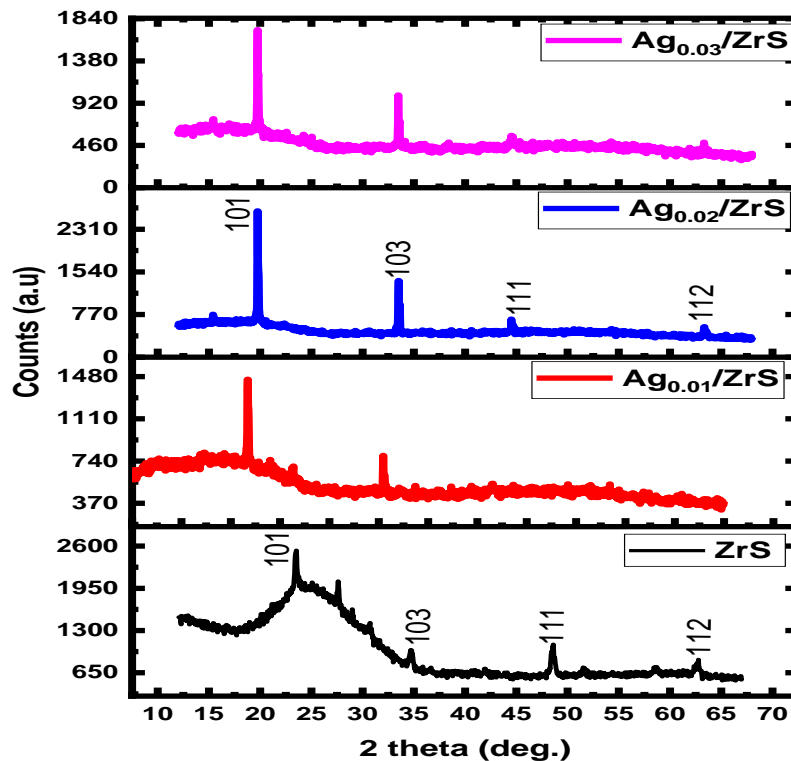
### Results and Discussion

#### The structural analysis of silver doped ZrS material.

The undoped zirconium sulphide (ZrS) and Ag/ZrS X-ray diffraction patterns (XRD) at dopant concentrations of 0.01 to 0.03 mol are shown in Figure 1. The ZrS and Ag/ZrS XRD patterns showed the materials are polycrystalline with distinct phase orientation planes. The intense peak, indexed at (101 to 112), represents values from 23.59° to 62.62°. It also has a heavily damped peak

plane at (111). The deposited material is appropriate for use in optoelectronics applications. (Chukwuemeka *et al.*, 2024; Emmanuel *et al.*, 2022; Akpu *et al.*, 2021; Ikhioya *et al.*, 2020; Ikechukwu and Ikhioya, 2024; Ikhioya *et al.*, 2020, 2021, 2023; Ikhioya and Ekpunobi, 2014a; Ikhioya and Nkele, 2023b, 2023a; Ikhioya *et al.*, 2023). When silver is incorporated into the ZrS lattice, it facilitates the formation of ZrS crystals. As the  $2\theta$  angle increases, the intensity of X-ray diffraction peaks increases, suggesting more crystallites and larger sizes. The increase in the size of the crystallites in Table 1 is causing the growth due to a decrease in surface energy. The total energy of the system decreases as the crystallites grow, leading to a decrease in the surface area. The addition of silver dopant atoms changes how crystals grow, leading to the formation of bigger crystallites. The larger crystallite size observed at higher  $2\theta$

angles in silver doped ZrS is due to crystal growth caused by reduced surface energy and the impact of silver dopant atoms on growth kinetics. Grain growth causes an increase in the crystallite size of silver-doped ZrS with an increasing  $2\theta$  angle. When the  $2\theta$  angle increases, the X-ray beam explores larger crystallites by penetrating deeper into the material. The X-ray diffraction pattern will reveal details about the larger crystallites, leading to increased peak intensity and broader peak width. A wider peak width suggests a greater range of crystal sizes, with the larger crystals causing the higher  $2\theta$  angles. In simpler words, as the X-ray beam goes deeper, it encounters bigger crystallites, causing the crystallite size to increase as the  $2\theta$  angle increases. This phenomenon occurs frequently in materials that demonstrate grain growth, where smaller crystallites combine to create larger ones.



**Figure 1: XRD pattern of ZrS and Ag/ZrS**

**Table 1: Structural parameters of ZrS and Ag/ZrS**

Film	2θ (deg.) Recast	d (spacing) Å	(Å)	(β)	(hkl)	(D) nm	σ lines/m <sup>2</sup>
ZrS	23.5941	3.7672	6.5250	0.2021	101	7.0076	6.2032
	34.9137	2.5674	5.1349	0.2021	103	7.1908	5.8912
	48.4287	1.8778	3.7557	0.2021	111	7.5215	5.3847
	62.6298	1.4819	3.3136	0.2021	112	8.0295	4.7250
Ag <sub>0.01</sub> /ZrS	19.6836	4.5059	7.8046	0.2032	101	6.9251	6.3518
	33.8347	2.6468	5.2936	0.2032	103	7.1318	5.9890
	44.5183	2.0332	4.0665	0.2032	111	7.3727	5.6042
	63.3158	1.4674	3.2814	0.2032	112	8.0163	4.7406
Ag <sub>0.02</sub> /ZrS	19.6836	4.5059	7.8046	0.2235	101	6.2961	7.6843
	33.8347	2.6468	5.2936	0.2235	103	6.4841	7.2453
	44.5183	2.0332	4.0665	0.2235	111	6.7031	6.7798
	63.3158	1.4674	3.2814	0.2235	112	7.2882	5.7351
Ag <sub>0.03</sub> /ZrS	19.6836	4.5059	7.8046	0.2300	101	6.1182	8.1377
	33.8347	2.6468	5.2936	0.2300	103	6.3009	7.6728
	44.5183	2.0332	4.0665	0.2300	111	6.5136	7.1798
	63.3158	1.4674	3.2814	0.2300	112	7.0823	6.0735

### Scanning electron microscopy (SEM) of ZrS and Ag/ZrS

Figure 2 displays the microstructure of Ag/ZrS. The micrograph of the ZrS material shows a presence of hexagonal structure material along with precipitate. The presence of various silver concentrations results in particle clumping and a uniformly coated substrate surface. The contact between the chalcogenide material and transition metal results in the formation of silver precipitate, and ZrS exhibits visible indications of material sublimation. SEM micrographs reveal the formation of diversely shaped nanoparticles, a consequence of the Ag/ZrS

reaction. The synthesized material, at a quantity of 0.01 mol, illustrates the transformation of the silver precipitate by the dopant, showing compatibility between the two materials. However, increasing the dopant molarity to (0.02 mol) caused the nanoparticles to restructure, showing that exceeding (0.03 mol) would cause material reversal. The XRD findings show that the rise in silver molarity directly correlates with the growth of crystallite size. The results show that film syntheses hold promise for solar cell and photovoltaic use. Figure 3 shows the elemental dispersive X-ray (EDXs) analyzes of the material. Zirconium, sulfur, and silver are the basic elements showed by the spectra.

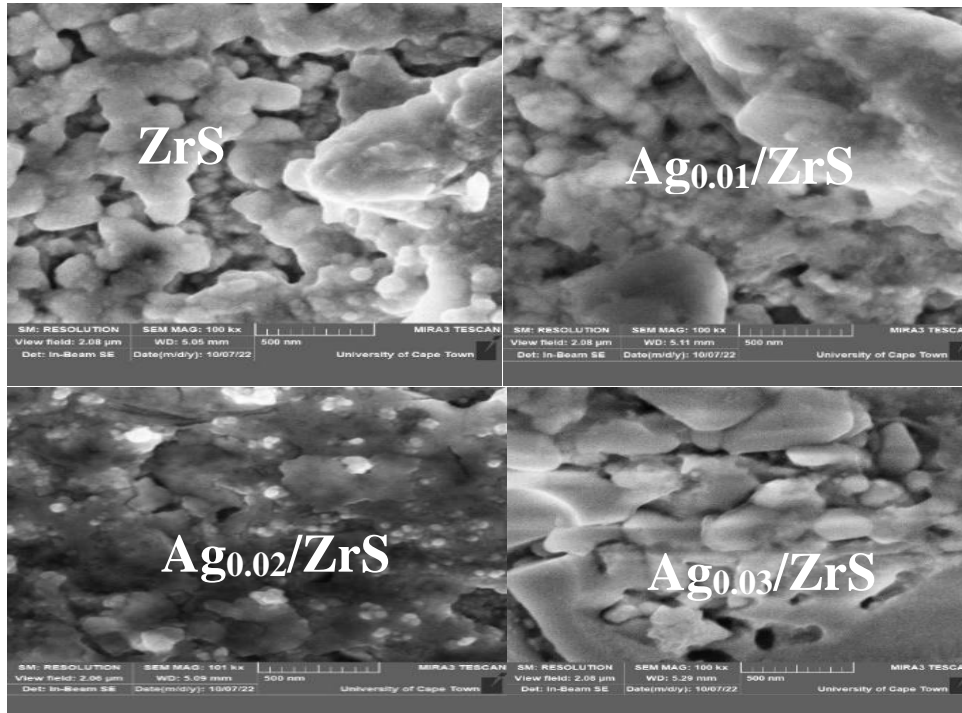


Figure 2: SEM micrograph of ZrS and Ag/ZrS

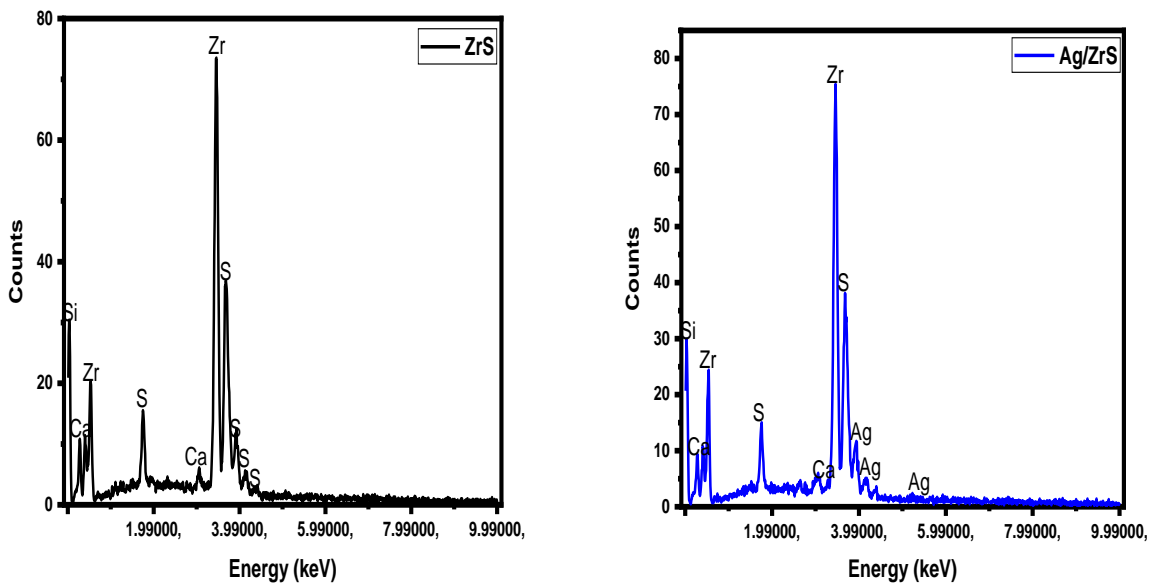


Figure 3: EDXs spectrum of ZrS and Ag/ZrS

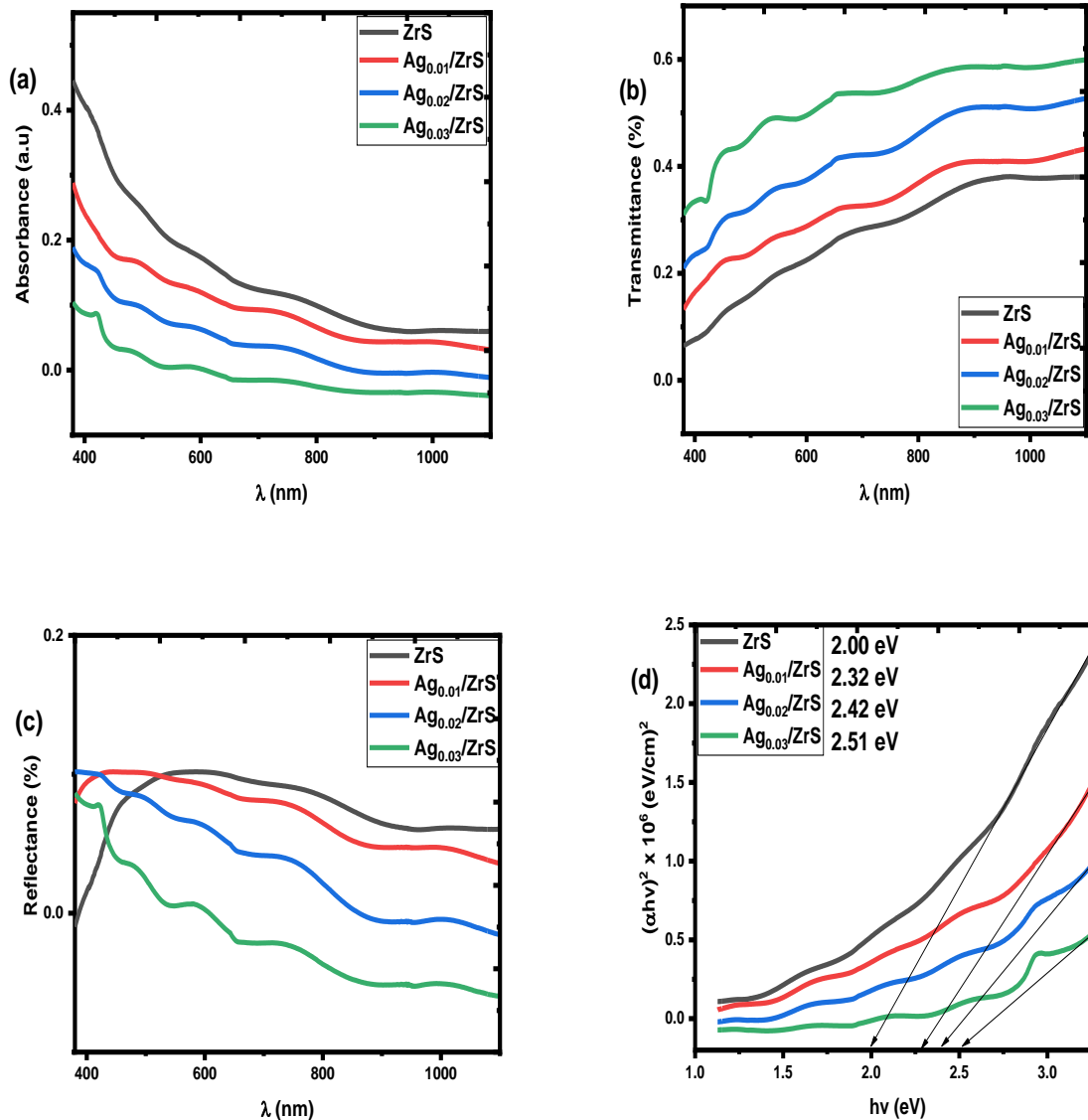
### Optical analysis of ZrS and Ag/ZrS

The absorbance of Ag/ZrS is shown in Figure 4 (a). As the wavelength of the films increases, the material's absorbance

decreases. In the visible region, the Ag/ZrS material displays a distinct absorption band, which is attributed to the silver surface plasmon resonance. The wavelength of the surface plasmon resonance band is

determined by the size, shape, and dielectric properties of the silver material and the surrounding ZrS material. Analyzing the absorption spectra allows for determining the properties of Ag/ZrS material, such as silver size and shape, ZrS dielectric properties, and material interaction. Ag/ZrS material finds applications in various fields like photocatalysis, solar cells, and sensors (Malumi *et al.*, 2023; Ojegu, Odia, *et al.*, 2023; Ojegu, Samuel, *et al.*, 2023; Okeoghene *et al.*, 2023; Rufus *et al.*, 2023; Samuel, Ojoba, *et al.*, 2023; Samuel, *et al.*, 2023; Sarwar *et al.*, 2023; Shah *et al.*, 2023; Udofia *et al.*, 2023; Udofia and Ikhioya, 2018). By examining the absorption spectra of Ag/ZrS material, its optical characteristics improve its functionality for specific uses. Figure 4 (b) displays the transmittance of Ag/ZrS. The material's transmittance increases as the wavelength of the films increases. The transmittance spectral measure the amount of light passing through a material at different wavelengths. By using this data, the material exhibits the properties for specific applications, such as optoelectronics or energy storage. The transmittance spectra of Ag/ZrS can design optoelectronic devices, such as solar cells, light emitting diodes (LEDs), and photodetectors. The efficiency and performance of these devices enhanced by customizing their optical properties. Figure 4 (c) displays the reflectance of Ag/ZrS. The material's reflectance decreases as the films' light radiation increases. The wavelength of incident light has a significant impact on the reflectance spectra of Ag/ZrS material. Ag/ZrS has strong reflectance in the visible region but decreases significantly in the near-

infrared region. The plasmonic resonance of silver in the Ag/ZrS composite is responsible for the behavior. The reflectance spectra of Ag/ZrS altered by manipulating the size and shape of silver. Smaller silver materials cause the plasmonic resonance peak to shift towards the blue end. The tunability of Ag/ZrS materials allows for designing specific optical properties for various applications. The application of Ag/ZrS materials with reflectance that varies with wavelength is versatile, spanning areas like photocatalysis, sensing, and optical devices. Ag/ZrS shows great potential for solar energy collection because it reflects light well in the visible region and has low reflectance in the near-infrared region, making it suitable for stealth technology. Figure 4 (d) displays the bandgap of Ag/ZrS. The Ag/ZrS material has a direct band gap at the UV point, making it ideal for optoelectronic applications. Efficient light emission happens when the valence band maximum and conduction band minimum align at the same k-point. The optoelectronic characteristics of Ag/ZrS material can be customized by varying the amount of Ag. By adjusting the Ag concentration, specific optical features can be achieved for different applications, altering the bandgap. The Ag/ZrS material has potential in optoelectronic applications as LEDs, photodetectors, and solar cells. Its potential to change its bandgap and efficient radiative recombination makes it a promising option for future optoelectronic devices (Kashif *et al.*, 2022). By incorporating silver into the ZrS lattice, the energy band structure rises from 2.32 to 2.51 eV as molarity increases.



**Figure 4: absorbance (a), transmittance (b), reflectance (c), and bandgap (d) of ZrS and Ag/ZrS**

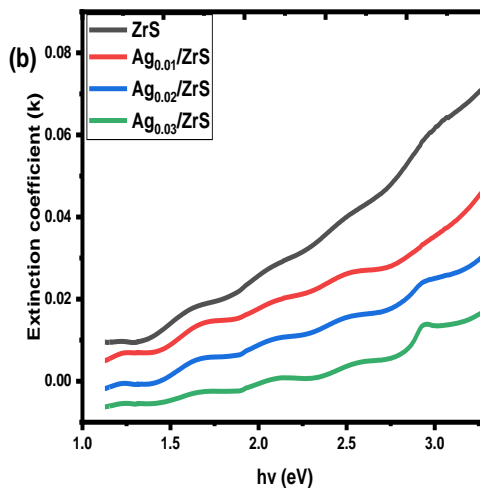
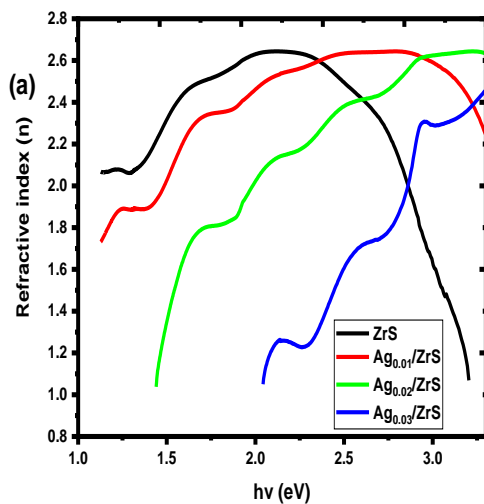
The refractive index of Ag/ZrS is illustrated in Figure 5 (a). As the photon energy of the material rises, the refractive index fluctuates. The refractive index spectra of Ag/ZrS material have important implications for its applications. For instance, in optoelectronics, the material's refractive index influences the performance of optical devices, such as lenses, prisms, and wave guides. Figure 5 (b) shows the extinction coefficient of Ag/ZrS.

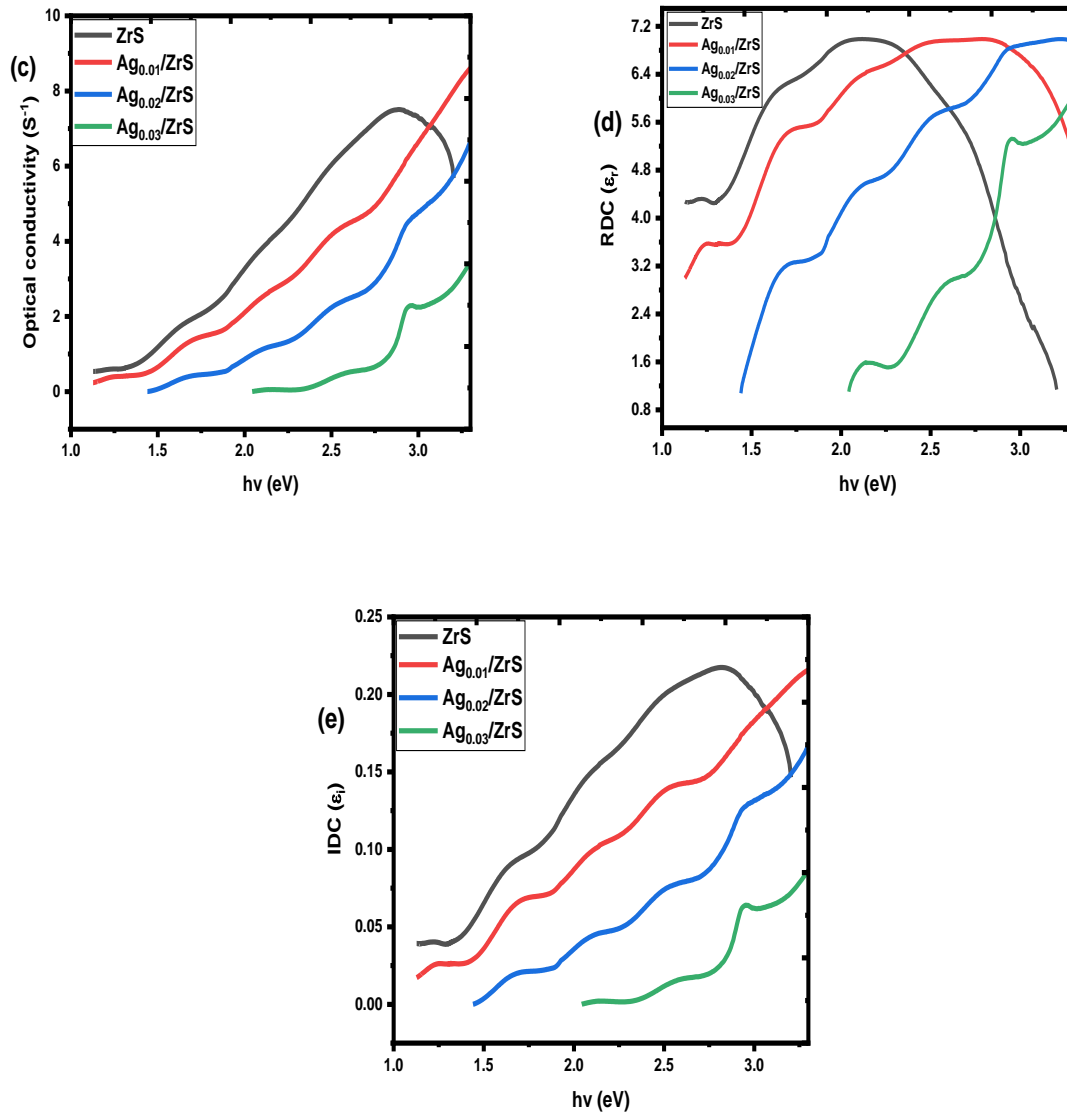
The increase in photon energy leads to an increase in the extinction coefficient of the material. The extinction coefficient quantifies how well a material can absorb and scatter light. By examining the spectra, scientists can learn about the material's uses in optoelectronics and other areas. Optimizing devices that use the optical properties of Ag/ZrS requires a thorough understanding of its extinction coefficient



spectra. The optical conductivity of Ag/ZrS is depicted in Figure 5 (c). As the photon energy rises, so does the material's optical conductivity. The optical conductivity spectra of Ag/ZrS show a clear dependence on photon energy. At low photon energies, the optical conductivity is dominated by intra-band transitions within the valence band. As the photon energy increases, inter-band transitions from the valence band to the conduction band become more significant, leading to a sharp increase in the optical conductivity. The optical conductivity of Ag/ZrS material increases with increasing photon energy. This is due to the increasing number of inter-band transitions that are possible in higher photon energies. The optical conductivity also shows a peak at around 2.3 eV, which corresponds to the bandgap of Ag/ZrS material. The optical conductivity spectra of Ag/ZrS material

study the electronic structure and optical properties of the material. Figure 5 (d&e) shows the dielectric constant (both real and imaginary parts) of Ag/ZrS. The real and imaginary dielectric constant of the material increases as the photon energy increases. The real dielectric constant measures a material's ability to store electrical energy, while the imaginary dielectric constant quantifies energy loss from polarization. Valuable information about the optical properties of Ag/ZrS material can be obtained by analyzing the photon energy spectra of these constants. The dielectric constant of Ag/ZrS material increases with higher photon energy. The material becomes more polarizable as the energy levels rise. As photon energy increases, the dielectric constant of Ag/ZrS material varies. At higher energies, the material encounters less energy loss due to polarization.





**Figure 5: refractive index (a), extinction coefficient (b), optical conductivity (c), real dielectric constant (RDC) (d), imaginary dielectric constant (IDC) (e) of ZrS and Ag/ZrS**

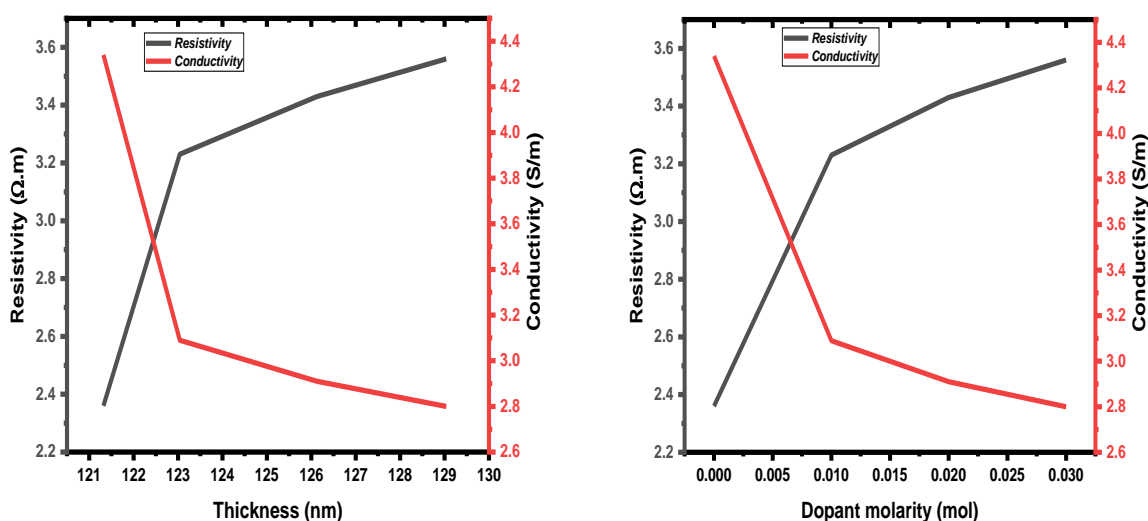
**The electrical analysis of ZrS and Ag/ZrS**

The resistivity and conductivity of ZrS and Ag/ZrS material are shown in Table 2. Increasing the film thickness from 121.32 to 129.04 nm resulted in a decrease in resistivity from 2.36 to 3.56 ohm/m and a drop in conductivity from 4.34 to 2.80 S/m. The films' low resistivity and conductivity make

them perfect for photovoltaic and solar cell applications. Figure 6 shows that as film thickness increases, resistivity decreases and conductivity increases. The graph shows the lack of an ohmic relationship between resistivity, conductivity, and silver molarity. The plot illustrates how film thickness, resistivity, conductivity, and silver molarity fluctuations are interconnected.

**Table 2: Electrical properties of ZrS and Ag/ZrS**

Film	t (nm)	$\rho$ ( $\Omega.m$ )	$\sigma$ ( $S/m$ ) <sup>-1</sup>
ZrS	121.32	$2.36 \times 10^6$	$4.34 \times 10^5$
Ag <sub>0.01</sub> /ZrS	123.04	$3.23 \times 10^6$	$3.09 \times 10^5$
Ag <sub>0.02</sub> /ZrS	126.13	$3.43 \times 10^6$	$2.91 \times 10^5$
Ag <sub>0.03</sub> /ZrS	129.04	$3.56 \times 10^6$	$2.80 \times 10^5$



**Figure 6: variation of resistivity and conductivity of ZrS and Ag/ZrS**

**Conclusions**

The successful utilization of the electrochemical deposition technique allowed for the synthesis and study of Ag/ZrS thin films. The XRD patterns of ZrS and ZrS/Ag indicated the materials are polycrystalline and have well-defined phase orientation planes. Increasing the 2 theta angle leads to stronger X-ray diffraction peaks, suggesting larger crystallites and greater crystallinity. Reduced surface energy drives the growth of the crystallites, as they increase in size. As the crystallites increase in size, the total energy of the system diminishes, resulting in a reduced surface area. Silver dopant atoms affect the way crystals grow, resulting in larger crystallites.

The increased crystallite size observed at higher 2 theta angles in silver-doped ZrS can be attributed to crystal growth, facilitated by lower surface energy and the impact of silver dopant atoms on growth kinetics. The resistivity dropped from 2.36 to 3.56 ohm/m as the film's thickness increased from 121.32 to 129.04 nm, resulting in a decrease in conductivity from 4.34 to 2.80 S/m. The films' low resistivity and conductivity make them perfect for photovoltaic and solar cell applications. By incorporating silver into the ZrS lattice, the energy band structure rises from 2.32 to 2.51 eV as molarity increases.

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#### Author contribution statement

**Shaka O. Samuel, Imosobomeh L. Ikhioya:** methodology, conceptualization, data curation, data collection, **Olisenekwu Cletus, Imosobomeh L. Ikhioya:** samples characterization, first-draft writing, software, reviewing, and editing. **Imosobomeh L. Ikhioya, Shaka O. Samuel:** supervisor, investigation and visualization. All authors approved the submission of the manuscript.

#### Disclosing conflicting interests.

The authors of this paper declare that there are no personal or financial conflicts that may have affected the study.

#### Availability of data

Access to the data is available upon request.

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