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Review Article

Synthesis and Characterization of Ag_2S Nano Crystalline Thin Films: A Review

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Abstract

Silver sulphide thin films have been grown using various deposition techniques. These techniques could be either physical or chemical deposition technique. A study of some properties (electrical, optical, structural, morphological, compositional) and their dependence on the preparation conditions presents a great important because it permits to establish the best growth conditions for obtaining films. The structure of the prepared films was investigated from X-ray diffraction pattern and the results indicate that the films were polycrystalline with acanthite structure. The refractive index was calculated in the visible region and the band gap value was varied in the range between 0.9 eV to 2.2 eV.

Keywords: Silver sulphide; Thin films; Chemical bath deposition; Solar cell; Semiconductor

Introduction

Synthesis and characterization of metal sulfide, metal selenide and metal telluride thin films have been investigated by many researchers. The experimental results were presented in many research articles and literature reviews as well [1-50]. Silver sulphide (Ag_2S) thin films belong to I-VI semiconductor compounds are blackish and show n-type electrical conductivity. These films have been studied for numerous applications such as solar cells, infrared detector, photoconducting cells and solar selecting coating, chemistry and biochemistry areas, due to their unusual optical, electrical and mechanical properties.

Currently, thin film solar cells are favorable due to their minimum material usage, thin, easy to handle and cheaper than silicon based solar cell. In the recent years, silver sulfide films have been prepared via various techniques such as hydrothermal method, thermal evaporation method, chemical bath deposition method, molecular beam epitaxy, spray pyrolysis method, ion beam deposition method, electro-deposition method, successive ionic layer adsorption and reaction method. However, researchers have mentioned that all these techniques have disadvantages and advantages, depend on the type of application intended for the films.

In this paper, preparation of silver sulphide films by using various deposition methods will be discussed.

Characterization of obtained films using different tools such as atomic force microscopy, x-ray diffraction, UV-visible spectroscopy, scanning electron microscopy, X-ray photoelectron spectroscopy and transmission electron microscopy will be reported.

Literature Survey

Preparation of silver sulphide films

$AgNO_3$ and Na_2S were used to prepare Ag_2S by using colloidal method at room temperature as reported by Jang et al. [51]. They explain that solutions of the different ions are mixed together under controlled temperature and pressure in order to produce insoluble precipitates. Hydrothermal deposition was used to prepare silver sulfide films in the presence of thiourea as described by Dong et al. [52]. There are several advantages of this method include the ability to produce films which are unstable near the melting point and the ability to prepare large crystals of high quality in aqueous medium. Ag_2S films were grown on glass and quartz substrates by thermal evaporation method as proposed by El-Nahass et al. [53]. They explain that this technique uses a strong vacuum environment and deposition rate is high, so that damage to the substrate during deposition process could be minimized and eventually produce high purity films. Maghraoui et al. [54] studied in detail the deposition of silver sulphide films using chemical bath deposition method from aqueous solutions of thiourea and silver nitrate, over a wide range of bath temperatures. This method is one of the cheapest methods because of it does not depend on expensive equipment to deposit thin films. Also, chemical bath deposited silver sulphide thin films were deposited on glass and polyester substrates using ammonia and sodium thiosulfate as complexing agent and a source of sulphur ion, respectively as pointed out by Ivan [55]. Molecular beam epitaxy method has many advantages such as it is particularly good for making high quality semiconductor thin films from compounds, thin films could be produced in a very precise and carefully controlled way. Monoclinic Ag_2S thin films were prepared onto cleaved surfaces of magnesium oxide (001) using molecular beam epitaxy as reported by Hiroshi et al. [56]. Thin films of silver sulphide have been prepared on Pyrex glass substrates by the spray pyrolysis method using silver acetate and thiourea as the starting materials as proposed by Dlala et al. [57]. This method possesses many advantages include low processing temperature, high homogeneity and purity of products. Simple and versatile the successive ionic layer adsorption and reaction method (SILAR) was employed to produce silver sulphide thin films onto different substrates such as glass and single crystalline wafer of silicon (111) as described by Sankapal et al. [58].

They explain that immersion of the substrate into separately placed silver nitrate (cation) and thiourea (anion) precursors solutions and rinsing (12 seconds) between every immersion with ion exchanged water to avoid homogeneous precipitation. Guo et al. [59] report the effect of applied potential on the properties of Ag_2S films grown by electro-deposition method. These films were deposited on indium tin oxide coated glass substrates in the presence of $AgNO_3$ and $Na_2S_2O_3$ with pH 2.5 at room temperature. The benefits of this method are thin film properties can be controlled and that processing can take place at room temperatures and pressures.

Characterization of silver sulphide films

Jang et al. [51] have proposed that back gate thin film transistor tool structure was produced on SiO_2/Si substrate in order to study the electrical properties of silver sulfide prepared using colloidal method. They confirm that the obtained films are n-type semiconductor and reveal the drain current of thin film transistor was increased based on the increase in the voltages of drain. The formation of cubic Ag_2S thin films prepared using simple hydrothermal route is confirmed by X-ray diffraction patterns as reported by Dong et al. [52]. They also highlight that the UV-visual absorption spectrum indicates obvious blue shift. The obtained thermally evaporated films are well crystallized according to the acanthite structure as indicated in X-ray diffraction studies as point out by El-Nahass et al. [53]. They also highlight some interesting results such as indirect allowed transition with a band gap of 0.96 eV, the dark electrical resistivity reduces with increasing film thickness, the values of lattice dielectric constant (7.8) and the ratio of the carrier concentration to the effective mass (1.7×1047 $kg^{-1}m^{-3}$).

The effect of bath temperature (40-80ºC) on the chemical bath deposited Ag_2S films was studied by Meherzi-Maghraoui et al. [54]. They conclude that the best deposition temperature was 60°C. The band gap measured was found to be in the range of 0.9 to 1.05 eV. Furthermore, they reveal that band gap was increased to 1.07 eV in annealed sample (at 250ºC for 60 min in nitrogen atmosphere). The uniform chemical bath deposited Ag_2S films were prepared by Ivan [55] at 50ºC and pH=10. Researcher notes that the absorption coefficient of the Ag_2S films was observed to reduce with increasing wavelength, indicating these films will be suitable for infrared detector applications. In addition, the resistivity was 14-20 Ω•m and the band gap about 2.2 eV as shown in experimental findings.

The epitaxial relations of the Ag_2S films to the substrates were studied by XRD as reported by Hiroshi et al. [56]. The X-ray diffraction (XRD) data confirm that three peaks are correspond to (012), (-112) and (040) planes. Dlala et al. [57] have reported the physical properties of sprayed Ag2S films obtained at various temperatures. It is seen from the XRD data that a strong peak appears at 2 theta=37.7° belonging to monoclinic structure. The compositional analysis was performed and the spectrum reveals the presence of silver (64.3%) and sulphur (32.7%) elements in the deposited films at 250°C.

Poor crystallinity and weak transmission could be seen in as-deposited thermally evaporated thin films as suggested by Agbo et al. [60]. However, annealing improved the crystallinity of the films and indicated acanthite structure with preferential orientation along (-121). The transmittance in the visible region is in the range of 45-55% and the band gap of the material varied from 2.1 to 2.2 eV. Thermally evaporated Ag_2S films were annealed at 250 ºC in argon atmosphere as described by Nasrallah et al. [61]. The obtained results showed that the Ag_2S annealed films have (103) preferential orientation and optical band gap of 1.1 eV. Microprobe analysis indicates that a nearly stoichiometric composition is seen in these materials.

SILAR deposition of silver sulphide thin films was carried out on various substrates by Sankapal et al. [58]. The XRD studies indicate that an improvement in crystallinity using Si (111) substrate as compared to that deposited on amorphous glass. Pathan et al. [62] have prepared silver sulphide thin films onto fluorine doped tin oxide coated glass substrates using this method also. They explain photovoltaic activity in polysulphide electrolyte by using cell configuration as Ag_2S/(1 M NaOH+1 M Na_2S+1 M S)/C at room temperature. The obtained films exhibit lower efficiency level (0.06%) due to nanocrystalline grain size and high value of series resistance.

Immersion cycles from 20,30,40 to 50 were studied by Kakade et al. [63] using SILAR method. The findings prove that with increasing the immersion cycle, peak intensity of (120) plane increases while the transmittance in the visible region and energy band gap was found to be reduced.

Good quality chemical bath deposited Ag_2S films were prepared using ethylene diamine tetra acetate disodium salt as complexing agent as described by Ezenwa et al. [64]. The micrograph indicated a uniform distribution of small grains over scanned area. These films have high absorbance in the ultra violet region and high refractive index (1.9-2.5 eV), make them important in photovoltaic technology. Starting materials such as $AgNO_3$ and $Na_2S_2O_3$ were used to produce silver sulfide films onto microscope glass substrate from a hot and cold solution in alkaline conditions (pH 8-11). X-ray diffraction patterns display that the high purity and film thickness could be controlled during the deposition process as suggested by Mimoza [65]. Further, researcher explains that the deposition rate increases with increasing alkalinity. Chemical deposition technique has been employed for the conversion of thin films of silver into Ag_2S films as described by Kulkarni et al. [66]. They demonstrate uniform and large area films were successfully synthesized onto glass substrates using the adsorption and reduction technique. There are five peaks correspond to (110), (120), (031), (200) and (102) are observed for the Ag_2S films prepared at room temperature in alkaline conditions. The presence of silver and sulphur in films was confirmed as displayed in energy dispersive X-ray analysis (EDAX) spectra. The surface roughness is 96.4 nm based on atomic force microscopy (AFM) image. The power conversion efficiency of films was 0.002% indicating high series resistance of films as pointed out by Jadhav et al. [67]. On the other hand, modified chemical bath deposition method has been used for the deposition of Ag_2S films as proposed by Jadhav et al. [68]. The bath contains ammonia, thiourea and silver nitrate. They conclude that the silver sulphide films had a maximum terminal thickness of 135 nm at 30 immersion cycles. Then, the film thickness was reduced because of the peeling off the outer powdery layer. They also confirm that the films have direct band gap (2.09 eV), average crystallite size (21 nm) and high absorbance value.

Silver rich and sulphur rich Ag_2S films were synthesized using ion beam deposition method and atmosphere & solution based sulfurization technique, respectively as suggested by Dias et al. [69]. They have reported that they were able to control the silver sulphide stoichiometry as shown in EDAX results under various sulfurization times and temperatures. Silver oxide, sulphur and polyformaldehyde were used as reactants during the deposition process to prepare Ag_2S films as suggested by Ye et al. [70]. The rod like nanoparticles with diameter of 0.1 µm and stoichiometry of Ag:S=1:0.453 could be found in transmission electron microscopy and energy dispersive X-ray analysis investigation, respectively.

The influence of different deposition potentials (-230 to -280 mV) on the electrodeposited Ag_2S thin films has been investigated by Guo et al. [59]. The monoclinic Ag_2S films with the relative deviation of cell parameters within 1.5% could be detected as shown in XRD studies. Meanwhile, the scanning electron microscopy results reveal that a uniform with better compactness can be obtained at more negative applied potential. The best potential is -250 mV, indicating the control of deposition potential lead to the control of the final properties of films.

Conclusion

This paper summarized the preparation of silver sulfide thin films by using different deposition methods. The properties of thin films that are important for applications are then addressed. The obtained films show acanthite structure, n-type semiconductor and direct band gap values lay in the range 0.9-2.2 eV.

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