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Compositional Analysis and Conductivity Enhancement of Annealed Chemically Deposited PbS Film

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Abstract: Compositional analysis using the energy dispersive X-ray Flourescence EDXRF' of chemically deposited lead sulphide (PbS) thin film from a bath ratio of 1:2 (Pb: Tu) is presented, deposition took place at room temperature from chemical bath constituted from Lead Acetate, Triethamolamine, Thiourea and Sodium Hydroxide. Conductivity type measurement on the as-prepared film showed a p-type conductivity with enhanced dark conductivity from 5-30 mA when annealed at 200°C for 1 h. The result emphasizes the fact that annealing at a predetermined temperature is essential to produce a good photoconductive PbS film.

Key words: Compositional analysis, chemical deposition, conductivity, annealed, photoconductivity, X-ray

INTRODUCTION

PbS is a member of the group of metal chalocogenides which are binary semiconductor compounds with combination of metals as cations and the chalocogenides sulphides slenides or telluride as anions. They are a group of semiconductor materials. For instance, the bandgap in metal chalcogenides may be controlled because their bandgaps diminish as their atomic number increases. Also, the temperature coefficients of bandgap for chalogenides of lead (PbS, PbSc and PbTe) are positive while they are negative for all other elemental or compound semiconductor materials. Their lattice structures may be very unstoichiometric, thereby creating vacancies and interstitials that can be exploited to control conductivity type. In fact, excess Pb atoms incline the Pb-chalogenides to n-type conductivity (Fajinmi *et al*., 2001).

Besides incorporating binary metalchalcogenide in multistructures, where they promote good applicability, they possess dynamic properties which present them as very good candidates in vast area of applications. For example, lead sulphide (PbS) thin film is found very suitable for near infrared (1-3 μm) sensors/detectors and as active layer in photothermal and photovoltaic applications. Chemically prepared very thin (20-50 μm) PbS films are also used for solar control coating and when carefully prepared such that the grains become quantum dots, whose sizes are comparable with the execution Bohr radius, PbS thin film is used as optical switch (Nair *et al*., 1989).

MATERIALS AND METHODS

The lead sulphide (PbS) thin films were deposited from aqueous chemical baths which were made up of 1 M Triethanolanine (TEA), with sufficient NaOH added to give a pH of 10-11 for the bath. For this research commercial glass slides (76×26×1 mm) were used as substrate. The glass slides were cleaned in detergent, chromic acid and distilled water and were supported vertically on the walls of 100 mL beakers for the chemical deposition. Compositional analysis was carried out using the energy dispersive X-ray fluorescence (Ed×Rfs) located in the center for Energy Research and Training (CERT), ABU, Zaria, Nigeria. The samples were put on the sample holder one after the other with he blank sample (the substrate) measured first. In order to record the photocurrent response, conducting silver paste paint was used to provide copper electrode and the film was then placed under 1KWm^{-2} Halogen lamp.

The bias voltage for the samples was supplied from an I.C. regulated variable power supply while the current flowing was measured with a digital multimeter.

The dark and photo-current were measured by the use of D.C. microameter. The photo-current response

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curve for the samples was recorded during a 500 sec period (100 sec in the dark, 200 sec under illumination and a further 200 sec in the dark). The PbS film (samples S_3) was annealed in an air oven at a temperature of 200°C for 1 h.

The thickness was determined by gravimetric estimation using known density of PbS.

RESULTS AND DISCUSSION

The deposition parameters of the PbS samples are given in Table 1.

The Table 1 shows there is an increase in thickness of the film with increase in deposition time. This is an expected result because as long as the bath is not depleted of constituent ions, deposition continues and thickness increases. There is also an increase in film thickness.

Table 2 and 3 present the results of the compositional analysis of the blank glass substrate and the PbS on substrate, respectively. In each table, the sample code of the equipment is given, the metric code (glass substrate in this case), is RES 1100 and the weight cm^{-2} as measured by a digital multimeter balance is also recorded. EL represents the element contained in the sample, E (KEV) is the energy at which each element is detectable, INT (C/S) is the intensity in counts per second of the detector source for each element, T is the geometry of each element and CONC (FRAC) is the fractional concentration of each element per sample. When the possible error in the measured concentration of an element is negligibly small and hardly detectable by the equipment, LOD low order of detection is recorded. The percent composition per thickness of lead (Pb) is calculated. This is done by subtracting the fractional concentration in the sample of the deposited film and finishing the percentage over the thickness of the sample.

The results on sample S_3 show that the PbS film has 65.27% lead and a trace of 0.57% Cu. The remaining 34.14% is believed to be sulphur since there is no trace of the presence of oxide in the film.

Figure 1 presents the photocurrent response of PbS film deposited from bath ratio 1:2 (Pb: Tu).

The drastic effect of annealing on the photo-current is clearly seen on it. It shows that the photo to dark current ratio decreased with increase in dark current.

This is in accordance with earlier results on PbS thin films by Nair and Nair (1990) and Fajinmi *et al.* (2001), where the photo to dark ratio decreased with increase dark and photocurrent as the film is annealed.

This is in agreement with the fact that annealing process is expected to heal defects in the PbS which may be crated during its deposition stage. This then allows for grain growth which results in conductivity increase.

The photo current response curves as presented in Fig. 2 show a phot-to-dark current ratio of only 4 or less under 1 KWm^{-2}, Tungsten halogen illumination.

Table 1: The deposition paramenters of the PbS

Sample	Deposition time	Deposition temp.	Mess of film g m^{-2}	Thickness of film (nm)
S_1	45 mn	28°C	1.80	0.39
S_2	1 h	28°C	1.95	0.52
S_3	2 h	28°C	0.254	0.7

$S_4 = S_3$ Annealed at 200°C for 1 h

Table 2: Results of the compositional analysis of the blank sample (substrate)

EL	E (KeV)	INT (c s^{-1})	S	T	Conc (Frac)	Error
K	3.312	0.052	1.65E+03	0.0025	1.18E-01	-LOD-
Ca	3.690	0.518	1.96E+03	0.0033	8.06E-01	-LOD-
Ti	3.390	0.056	4.50E+03	0.0042	2.91E-03	-LOD-
Ba	4.508	0.057	0.09E+03	0.0041	6.59E-03	-LOD-
V	4.450	0.039	6.74E+03	0.0054	1.04E-03	-LOD-
Cr	4.949	0.042	8.76E+03	0.0067	7.03E-04	-LOD-
Mn	5.411	0.050	1.11E+04	0.0083	5.26E-03	-LOD-
Fe	5.895	0.222	1.38E+04	0.0103	1.53E-03	-LOD-
Co	6.400	0.041	1.71E+04	0.0126	1.88-04	-LOD-
Ni	6.925	0.033	2.14E+04	0.0153	9.78E-05	-LOD-
Cu	7.472	0.034	2.51E+04	0.0185	7.21E-01	-LOD-
Zn	8.041	0.042	3.01E+04	0.0221	6.21E-05	-LOD-
As	8.631	0.055	4.27E+04	0.0361	3.50E-05	-LOD-
Pb	10.532	0.047	2.50E+04	0.0480	5.05E-01	-LOD-
Rb	10.540	0.037	5.93E+04	0.0619	1.28E-05	-LOD-
Sr	11.907	0.070	6.46E+04	0.0695	1.72E-05	-LOD-
Y	13.375	0.337	7.03E+04	0.0775	6.75E-05	-LOD-
Zr	14.142	0.043	7.60E+04	0.0858	7.09E-06	-LOD-
Nb	14.933	1.213	9.77E+04	0.0942	1.42E-04	-LOD-
Mo	14.746	0.047	1.06E+05	0.1029	4.63E-06	-LOD-

Sample: IS528 Matrix: [AO (RES) = 1100], Weight [g m^{-2}] 0.150

Table 3: Result of the composition analysis of sample

EL	E (KeV)	INT (c s^{-1})	S	T	Conc (Frac)	Error
K	3.312	0.029	4.77E+02	0.0026	S	-LOD-
Ca	3.690	0.141	5.67E+03	0.0032	2.95	-LOD-
Ti	4.508	0.035	1.30E+03	0.0040	8.65	-LOD-
Ba	4.088	0.029	1.05E+03	0.0032	7.76	-LOD-
V	4.949	0.028	1.95E+03	0.0051	2.91	-LOD-
Cr	5.411	0.032	2.54E+03	0.0064	1.04	-LOD-
Mn	5.895	0.027	3.23E+03	0.0079	7.04	-LOD-
Fe	6.400	0.112	3.99E+03	0.0098	5.26	-LOD-
Co	6.925	0.028	4.95+03	0.0120	2.78	-LOD-
Ni	7.472	0.026	6.19E+03	0.0145	1.88	-LOD-
Cu	8.041	0.040	7.28E+03	0.0174	7.25	-LOD-
Zn	8.631	0.027	8.73E+03	0.0208	2.80	-LOD-
As	10.532	0.052	1.24E+04	0.0390	2.71	-LOD-
Pb	11.210	0.032	1.39E+04	0.0338	9.62	-LOD-
Rb	10.462	0.154	4.05E+04	0.0542	3.75	-LOD-
Sr	11.907	0.028	1.10E+04	0.0567	2.72	-LOD-
Y	14.142	0.033	1.34E+04	0.0696	3.96	-LOD-
Zr	14.933	0.036	3.04E+04	0.0759	5.84	-LOD-
Nb	13.600	0.055	3.32E+04	0.0821	4.63	-LOD-
Mo	15.746	0.074	2.87E+05	0.0882	3.98	-LOD-

IS8030 Matrix [AO(RES) = 1100 Weight [g m^{-2}]: 0.254

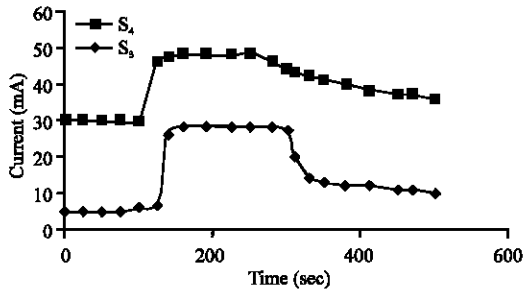

Fig. 1: Photocurrent response of PbS film for samples S_3 and S_4

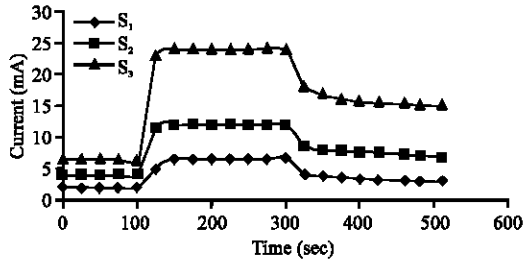

Fig. 2: Photocurrent response curves of sample S_1, S_2 and S_3 showing photodark current ratio (of 4 or less) under 1 kWm^{-2} tungsten halogen illumination

The dark current shows some increase with increase in thickness from 2 mA for a film of 0.39 mn thickness (Sample S_1) to 6 mA for a film of 0.07 nm in thickness (Sample S_3). This is a common feature of thin films arising from charge carrier trapping at the surface and inter-grain boundaries, which are thickness dependent (Orton *et al*., 1982; Kazmerski, 1980).

CONCLUSION

This study has demonstrated the case of depositing good quality PbS thin films from chemical baths. The compositional analysis using the energy dispersive X-ray fluorescence revealed that the as prepared film of thickness 0.7 nm contained about 65.27% lead and 34.14% sulphur with about 0.57% trace of copper. The result show an increase in dark current of about 24 mA and an increase in photocurrent of about 21 mA for the 0.7 nm thick film when annealed at 200°C for 1 h.

The results in this research suggest that there exists the possibility of modifying the film properties considerably by choosing suitable bath ratios of (Pb: Tu) ad multilayer structures which could be deposited from chemical baths.

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