Research Article

Compositional Dependence of Structural Properties of Prepared $\text{Pb}_x\text{S}_{1-x}$ Alloys and Films

M. F. A. Alias,¹ A. A. J. Al-Douri,² E. M. N. Al-Fawadi,¹ and A. A. Alnajjar²

¹ Department of Physics, College of Science, University of Baghdad, P.O. Box 47162, Jadiriya, Baghdad, Iraq

² Department of Applied Physics, College of Sciences, University of Sharjah, P.O. Box 27272, Sharjah, UAE

Correspondence should be addressed to A. A. J. Al-Douri, douri@sharjah.ac.ae

Received 21 May 2011; Revised 15 August 2011; Accepted 22 August 2011

Academic Editor: Rosa Lukaszew

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Results of a study of $\text{Pb}_x\text{S}_{1-x}$ alloys and films with various Pb content have been reported and discussed. Films of $\text{Pb}_x\text{S}_{1-x}$ of thickness $1.5\text{ }\mu\text{m}$ have been deposited on glass substrates by flash thermal evaporation method at room temperature, under vacuum at constant deposition rate. These films were annealed under vacuum around 10^{-6} Torr at different temperatures up to 523 K. The composition of the elements in $\text{Pb}_x\text{S}_{1-x}$ alloys was determined by standard surfaces techniques such as atomic absorption spectroscopy (AAS) and X-ray fluorescence (XRF), and the results were found of high accuracy and in very good agreement with the theoretical values. The structure for alloys and films is determined by using X-ray diffraction. This measurement reveals that the structure is polycrystalline with cubic structure and there are strong peaks at the direction (200) and (111). The effect of heat treatment on the crystalline orientation, relative intensity, and grain size of $\text{Pb}_x\text{S}_{1-x}$ films is presented.

1. Introduction

Long-wavelength infrared material systems are of great importance for many industrial applications [1–3]. Accordingly material systems at this wavelength region have been extensively studied for the last four decades.

Lead sulfide (PbS) is one of early materials used at this wavelength region since its properties are suitable for numerous civilian applications. PbS has a rock-salt structure [4], so it has cubic lattice with unit cell face-centered cube (FCC) with lattice constant ($a = 5.94\text{ }\text{\AA}$) [5–8]. PbS has a narrow optical energy gap in the near-infrared region [9]. This spectral range is of great interest for various optical and photo-thermal conversion applications [2, 3, 10, 11].

In this work $\text{Pb}_x\text{S}_{1-x}$ alloys were prepared with different x content: 0.50, 0.51, 0.52, 0.54, and 0.55. A new approach was implemented for fabricated $\text{Pb}_x\text{S}_{1-x}$ films using vacuum flash evaporation technique.

The properties of this new system of $\text{Pb}_x\text{S}_{1-x}$ films, such as the electrical, electronic, optical, and photoelectrical properties, had already been investigated [12, 13]. The aim of this work is to study the role of x content and annealing temperature on the structural properties of this system as a bulk and films.

2. Experimental

The bulk material was prepared by using the well-known melt quenching method, in which highly pure lead and sulfur elements (99.9999%) were molted to obtain $\text{Pb}_x\text{S}_{1-x}$ alloys with different x content where x (0.50, 0.51, 0.52, 0.54, and 0.55). This alloy was used as a source of the evaporation material to prepare the films. Thin film samples having thickness a round $1.5\text{ }\mu\text{m}$ were deposited on clean glass substrates at room temperature (RT) using flash evaporation technique. The Edward Coating Unit (E306A) was employed as a vacuum chamber under pressure 10^{-6} Torr during deposition. The deposition rate was kept nearly constant around $(0.83 \times 10^{-3}\text{ }\mu\text{m/sec})$, and the film thickness was controlled using Fizeau fringes interferometer employed at sodium wavelength with thickness accuracy $\pm 0.15\text{ }\mu\text{m}$. The prepared samples were unannealed (RT) and annealed under vacuum ($\sim 10^{-5}$ Torr) for one hour in the range (373–523) K. These samples were used to assess the effect of annealing temperature on the structure of $\text{Pb}_x\text{S}_{1-x}$ thin films.

The composition of $\text{Pb}_x\text{S}_{1-x}$ alloys was determined by Shimadzu 601 instrument atomic absorption spectroscopy (AAS). This analysis was used for quantitative measurement of atomic percentage of the material (Pb and S) in the alloys.

Also, X-ray fluorescence (XRF) has been used to determine the composition analysis of these alloys. The structure of the $\text{Pb}_x\text{S}_{1-x}$ alloys as bulks and films has been examined by X-ray diffraction (XRD), with CuK_α wavelength (λ) (1.5405 Å). The inter planer distance d (hkl) for different planes was measured using Braggs law [14]. The lattice constant (a) were estimated employing conventional method [15] which can calculate by using

$$a = \frac{d}{(h^2 + k^2 + l^2)^{1/2}}, \quad (1)$$

where d is interplaner distances for different phases which are measured by using Braggs law [16]

$$n\lambda = 2d \sin \theta, \quad (2)$$

where n is the reflection order and (hkl) is miller indices. The rough estimate of the PbS grain size dimension (D) was evaluated from diffraction line broadening using the Scherrer equation [17, 18]

$$D = 0.9 \frac{\lambda}{B \cos \theta}, \quad (3)$$

where λ is the X-ray wavelength, B is line broadening, that is, the width of PbS diffraction line at half maximum intensity, and θ is the diffraction angle.

3. Results and Discussion

3.1. Composition. Atomic absorption spectroscopy (AAS) has been used to examine the concentration of the elements (Pb and S) in the alloys as bulks. The atomic absorption of the standard elements of these components was taken as standard reference in order to obtain high accuracy in measuring the concentration of the elements which constitute the alloys. Confirmation of these measurements was determined using XRF technique. We observed that the two values are very close and in good agreement with theoretical values as shown in Table 1. These results indicate that this method is adequate for producing homogenous composition of $\text{Pb}_x\text{S}_{1-x}$ alloys.

3.2. Structural Analysis. Figure 1 shows the X-ray diffraction for $\text{Pb}_x\text{S}_{1-x}$ alloys with x content (0.50, 0.51, 0.52, 0.54, and 0.55) it appears that these alloys as bulks are polycrystalline of FCC structure according to ASTM cards. Analysis of the experimental data was done using reference of the intensities listed for the reflection of the PbS cards. The interplaner distances (d_{hkl}) for different planes were measured and compared with the ASTM cards data for FCC of $\text{Pb}_x\text{S}_{1-x}$ alloys [19]. The reflection surface [(111), (200), (220), (311), (222)] has been appeared as shown in Figure 1, and it is similar to the ASTM cards data. This figure shows crystallite with strong peak at (200) direction, and the intensity of other planes decreases with increasing Pb content for alloys. Figures 2(a)–2(c) shows the X-ray patterns for unannealed $\text{Pb}_x\text{S}_{1-x}$ and annealed at both temperatures (373, 523 K) and different x content it appears that all prepared and annealed

FIGURE 1: X-ray diffraction pattern for $\text{Pb}_x\text{S}_{1-x}$ alloy at various value of x content.

FIGURE 2: X-ray diffraction pattern for $\text{Pb}_x\text{S}_{1-x}$ films at various x content and (a) unannealed (RT) (b) $T_a = 373$ K (c) $T_a = 523$ K.

TABLE 1: The composition of alloys determined by AAS and XRF techniques.

Alloy	Theoretical concentration of element		Experimental concentration of element by AAS		Experimental concentration of element by XRF		Total
	Pb	S	Pb	S	Pb	S	
Pb _{0.50} S _{0.50}	0.50	0.50	0.50	0.50	0.500	0.500	1.0
Pb _{0.51} S _{0.49}	0.51	0.49	0.490	0.510	0.510	0.490	1.0
Pb _{0.52} S _{0.48}	0.52	0.48	0.520	0.480	0.520	0.480	1.0
Pb _{0.54} S _{0.46}	0.54	0.46	0.550	0.450	0.547	0.453	1.0
Pb _{0.55} S _{0.45}	0.55	0.45	0.555	0.445	0.550	0.450	1.0

TABLE 2: X-ray diffraction data for Pb_xS_{1-x} alloys as bulks.

Alloy	<i>hkl</i>	(<i>I/I₀</i>) _{stand}	<i>d</i> _{stand} (Å)	2θ	<i>d</i> (Å)	<i>a</i> (Å)	<i>D</i> μm x10 ⁻¹	(<i>I/I₀</i>)
Pb _{0.50} S _{0.50}	111	84	3.429	26.15	3.404	5.900	0.407	100.00
	200	100	2.696	30.20	2.958	5.920	0.411	100.00
	220	57	2.099	43.20	2.092	5.920	0.427	75.00
	311	35	1.790	51.175	1.783	5.914	0.440	55.00
	222	16	1.714	53.575	1.709	5.92	0.445	35.00
Pb _{0.51} S _{0.49}	111	84	3.429	26.20	3.400	5.900	0.329	55.00
	200	100	2.969	30.20	2.960	5.920	0.326	100.00
	220	57	2.099	42.80	2.108	5.960	0.341	48.40
	311	35	1.790	50.6	1.8	5.96	0.263	27.60
	222	16	1.714	52.9	1.72	5.95	0.266	24.00
Pb _{0.52} S _{0.48}	111	84	3.429	25.70	3.451	5.970	0.244	78.00
	200	100	2.969	30.00	2.973	5.946	0.282	100.00
	220	57	2.099	42.80	2.106	5.950	0.256	22.00
	311	35	1.790	50.8	1.792	5.94	0.211	15.00
	222	16	1.714	53.2	1.717	5.947	0.266	16.00
Pb _{0.54} S _{0.46}	111	84	3.429	25.60	3.470	6.000	0.325	35.00
	200	100	2.969	30.20	2.960	5.920	0.246	100.00
	220	57	2.099	42.10	2.140	6.000	0.340	11.00
Pb _{0.55} S _{0.45}	111	84	3.429	25.60	3.470	6.010	0.326	15.28
	200	100	2.969	30.00	2.973	5.946	0.246	100.00

films are polycrystalline of FCC structure. One observed from these data that, a strong peak at (111) direction for films annealed at 373 and 523 K, but its intensity decreases for other planes with increasing Pb content as shown in Figures 2(b) and 2(c) except at the peak (222) annealed at 373 K this means that the planes (200) and (111) are grown with crystal growth for alloy as bulks and films. Similar observations have also been obtained by other researchers [20–24], whereas for unannealed film the strong peak is at (200).

The increase of lead content and T_a improves the crystal structure by decreasing the intensity of the planes (220), (311), (222), while it increased for alloys as bulks and unannealed films in the direction (200), and in the direction (111) for films annealed at 373 and 523 K. These data also shows a little shifting toward higher diffraction angle with increasing x content and T_a . Therefore Pb_xS_{1-x} alloys as bulks have tendency preferential with increasing x content and T_a towards the plane (200) and (111) crystallite orientation. Such improvement in crystal structure could be attributed to the increase in crystallite size as the small crystallites join each other in the planes (200) and (111). The data also shows that the main changes in Pb_xS_{1-x} crystal structure films are observed after annealed to 373 and 523 K. This is in agreement with outcomes of other studies [20, 22, 25]. The lattice constant (a) is nearly equal to the standard value (5.94 Å) and varies around this value with increasing annealing temperatures and x content as shown in Table 2.

It could be seen from Figures 1 and 2 that the peaks for alloys and films which have smaller intensities (222), (311), (220) show the tendency towards decreasing the relative intensity as well as decrease in (FWHM), and that leads to increase in crystalline size. This is attributed to recrystallization of Pb_xS_{1-x}, since the increasing in lead content leads to improve the crystal structure. The relative intensity corresponding to the planes (111) and (200) as a function of the amount of x content in the alloys as illustrated in Figure 1 and for the films as shown in Figure 2, clearly indicates that the increasing Pb content gradually promotes orientation of the (111) and (200) directions. The value of I/I_0 shows always higher than the reference value and higher than the value obtained for other researchers [17, 20, 21]. The relative intensities observed for alloys as bulks and films with various of x generally do not differ from the listed values for PbS materials standard but give higher relative intensities of the reflection in the (111) and (200) direction, and this is in agreement with other studies [20, 25]. Finally the addition of Pb in the alloys as bulks and films will gradually orient the alloys as bulks in the (111) and (200) direction, respectively. Our experiments also showed that the final degree of orientation is strongly affected by the change in x content.

The crystallite dimension, grain size, (D) for the alloys as bulks unannealed and annealed films at 373 and 523 K are tabulated in Tables 2 and 3, respectively. The minimum and

TABLE 3: X-ray diffraction data for (a) unannealed (RT) and annealed $\text{Pb}_x\text{S}_{1-x}$ films (b) $T_a = 373 \text{ K}$ (c) $T_a = 523 \text{ K}$.

(a)								
Film	hkl	$(I/I_o)_{\text{stand}}$	$d_{\text{stand}}(\text{\AA})$	2θ	$d(\text{\AA})$	$a(\text{\AA})$	$D \mu\text{m} \times 10^{-1}$	(I/I_o)
$\text{Pb}_{0.50}\text{S}_{0.50}$	111	84	3.429	26.150	3.404	5.895	0.362	100.00
	200	100	2.696	30.175	2.959	5.918	0.821	100.00
	220	57	2.099	43.200	2.092	5.917	0.341	90.36
	311	35	1.790	51.175	1.783	5.914	0.439	60.24
	222	16	1.714	53.575	1.709	5.920	0.395	28.30
$\text{Pb}_{0.51}\text{S}_{0.49}$	111	84	3.429	26.400	3.363	5.825	0.392	74.10
	200	100	2.969	30.600	2.917	5.834	0.244	100.00
	220	57	2.099	43.500	2.077	5.875	0.256	96.00
	311	35	1.790	51.400	1.774	5.884	0.360	65.48
	222	16	1.714	53.800	1.701	5.892	0.268	40.00
$\text{Pb}_{0.52}\text{S}_{0.48}$	111	84	3.429	26.500	3.348	5.800	0.329	73.40
	200	100	2.969	30.600	2.911	5.820	0.219	100.00
	220	57	2.099	43.600	2.071	5.860	0.256	83.40
	311	35	1.790	51.600	1.768	5.864	0.423	60.00
	222	16	1.714	54.000	1.695	5.872	0.237	36.00
$\text{Pb}_{0.54}\text{S}_{0.46}$	111	84	3.429	26.150	3.404	5.895	0.408	100.00
	200	100	2.969	30.220	2.958	5.916	0.329	100.00
	220	57	2.099	43.200	2.092	5.917	0.427	88.55
	311	35	1.790	51.180	1.783	5.914	0.440	58.43
	222	16	1.714	53.575	1.709	5.920	0.418	26.50
$\text{Pb}_{0.55}\text{S}_{0.45}$	111	84	3.429	26.150	3.404	5.895	0.435	85.20
	200	100	2.969	30.175	2.959	5.918	0.328	100.00
	220	57	2.099	43.200	2.092	5.917	0.427	74.60
	311	35	1.790	51.165	1.783	5.914	0.440	30.00
	222	16	1.714	53.475	1.712	5.937	0.444	21.18
(b)								
Film	hkl	$(I/I_o)_{\text{stand}}$	$d_{\text{stand}}(\text{\AA})$	2θ	$d(\text{\AA})$	$a(\text{\AA})$	$D \mu\text{m} \times 10^{-1}$	(I/I_o)
$\text{Pb}_{0.50}\text{S}_{0.50}$	111	84	3.429	25.3	3.504	6.060	0.244	45.48
	200	100	2.696	29.4	3.020	6.040	0.246	35.68
	220	57	2.099	43.8	2.063	5.835	0.291	25.80
	311	35	1.790	50.5	1.804	5.980	0.263	17.60
	222	16	1.714	52.9	1.727	5.980	0.532	15.30
$\text{Pb}_{0.51}\text{S}_{0.49}$	111	84	3.429	25.9	3.426	5.930	0.332	100.00
	200	100	2.969	30.1	2.960	5.920	0.246	93.00
	220	57	2.099	43.1	2.095	5.925	0.256	50.90
	311	35	1.790	51.5	1.783	5.910	0.264	34.60
	222	16	1.714	53.4	1.711	5.927	0.266	26.70
$\text{Pb}_{0.52}\text{S}_{0.48}$	111	84	3.429	25.9	3.431	5.940	0.245	100.00
	200	100	2.969	30.1	2.963	5.926	0.247	98.50
	220	57	2.099	43.0	2.100	5.939	0.256	49.00
	311	35	1.790	51.0	1.786	5.923	0.213	31.70
	222	16	1.714	53.5	1.709	5.920	0.267	32.60
$\text{Pb}_{0.54}\text{S}_{0.46}$	111	84	3.429	26.0	3.420	5.920	0.282	77.64
	200	100	2.969	30.1	2.961	5.922	0.247	100.00
	220	57	2.099	43.0	2.098	5.934	0.256	45.08
	311	35	1.790	51.0	1.788	5.930	0.264	36.10
	222	16	1.714	53.4	1.710	5.920	0.307	22.34
$\text{Pb}_{0.55}\text{S}_{0.45}$	111	84	3.429	25.9	3.430	5.940	0.139	100.00
	200	100	2.969	29.9	2.980	5.960	0.246	38.40
	220	57	2.099	42.8	2.108	5.960	0.341	20.78
	311	35	1.790	50.9	1.791	5.940	0.232	21.16
	222	16	1.714	53.4	1.713	5.934	0.267	66.26
(c)								
Film	hkl	$(I/I_o)_{\text{stand}}$	$d_{\text{stand}}(\text{\AA})$	2θ	$d(\text{\AA})$	$a(\text{\AA})$	$D \mu\text{m} \times 10^{-1}$	(I/I_o)
$\text{Pb}_{0.50}\text{S}_{0.50}$	111	84	3.429	25.9	3.430	5.940	0.277	100.00
	200	100	2.696	30.0	2.973	5.946	0.279	43.70
	220	57	2.099	42.9	2.101	5.942	0.256	36.20
	311	35	1.790	50.9	1.79	5.936	0.211	24.88
	222	16	1.714	53.1	1.719	5.95	0.195	23.3

(c) Continued.

Film	hkl	$(I/I_0)_{\text{stand}}$	$d_{\text{stand}}(\text{\AA})$	2θ	$d(\text{\AA})$	$a(\text{\AA})$	$D \mu\text{m} \times 10^{-1}$	(I/I_0)
Pb _{0.51} S _{0.49}	111	84	3.429	25.7	3.451	5.970	0.244	78.80
	200	100	2.969	30.0	2.973	5.946	0.197	33.00
	220	57	2.099	42.8	2.106	5.956	0.289	22.00
	311	35	1.790	50.8	1.792	5.943	0.351	15
	222	16	1.714	53.2	1.717	5.947	0.266	16.5
Pb _{0.52} S _{0.48}	111	84	3.429	25.8	3.442	5.961	0.195	100.00
	200	100	2.969	29.8	2.993	5.986	0.279	21.60
	220	57	2.099	42.8	2.108	5.960	0.341	7.80
	311	35	1.790	50.7	1.797	5.95	0.264	9.4
	222	16	1.714	53.2	1.718	5.95	0.266	41.2
Pb _{0.54} S _{0.46}	111	84	3.429	26.0	3.421	5.930	0.196	100.00
	200	100	2.969	30.0	2.267	5.934	0.341	25.08
	220	57	2.099	42.9	2.101	5.942	0.256	9.00
	311	35	1.790	50.9	1.791	5.94	0.264	12.14
	222	16	1.714	53.4	1.711	5.93	0.267	40.78
Pb _{0.55} S _{0.45}	111	84	3.429	25.8	3.440	5.958	0.407	100.00
	200	100	2.969	29.8	2.993	5.988	0.411	17.40
	220	57	2.099	42.8	2.108	5.962	0.426	31.20
	311	35	1.790	50.7	1.797	5.959	0.292	12
	222	16	1.714	53.2	1.718	5.95	0.222	10

maximum values of crystallite size for these alloys as bulks and films are 20 nm and 82 nm, respectively. The range of crystallite size reported by other researchers varies from (10–2000) nm depending on the method of preparation [22]. In general, it was nearly observed that crystallite size alters with increasing x content and T_a as shown in Tables 2 and 3, and this is attributed to the recrystallization of the Pb _{x} S_{1– x} which takes place when we added excess lead. Judita [20] has calculated the grain size of PbS films which deposited on Si substrate by using reaction method, and she found that the grain size varies between (55–60) nm with increasing annealing temperatures.

4. Conclusions

The Pb _{x} S_{1– x} alloys for $0.55 \leq x \leq 0.50$ have successfully been prepared, and films were fabricated at different evaporation conditions using flash thermal evaporation technique. These films were annealed at different temperatures under a vacuum. From our data we concluded that

- (i) the concentration of Pb content for Pb _{x} S_{1– x} alloys found very close to the theoretical values;
- (ii) the structure of prepared alloys and films was polycrystalline with FCC;
- (iii) the crystalline orientation, relative intensity, and grain size of Pb _{x} S_{1– x} films were affected by heat

treatment within the range (373–523) K and with preferred orientation, (200) and (111) orientation.

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