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Structural Properties of PbInS Thin Films Prepared with Different Volume Ratio

Alfind Paul Frit. A, Deepalakshmi. K, Jothi. S, Alagulakshmi. A, Prithivikumaran. N, Jeyakumaran. N*
Energy Physics Lab, Department of physics, V.H.N.S.N. College (Autonomous),
Virdhunagar-626 001, Tamilnadu, India.

***Abstract*— The Lead Indium Sulphide (PbInS) semiconductor thin films were deposited on porous silicon substrate using dip coating method. The X-ray diffraction (XRD) patterns confirmed the polycrystalline structure of PbInS phase with preferential orientation along (7 0 1) plane. The crystallite size of the thin films varied from 102 to 80 nm with respect to volume ratio changes of annealing temperature at 400°C. Scanning electron microscope images showed that all the films have uniform surface morphology over the entire substrate and that the films were of good quality.**

Keywords*— Porous silicon; PbS powder; InS powder; PbInS film

I. INTRODUCTION

The ever-increasing interest into deposition of ternary derivative materials is due to the potential of designing and tailoring both the lattice parameters and the forbidden band-gap energy (E_g) by controlling growth parameters. This material has also been used in many fields such as photography, Pb^{2+} ionselective sensors and solar absorption [1]. In addition, PbS has been utilized as photoresistance, diode lasers, humidity and temperature sensors, decorative and solar control coatings [2]. Later on PbS thin films have been considered for their suitability for photothermal and thermophotovoltaic conversion of solar energy [3]. Indium sulfide (In_2S_3) is an important material for optoelectronic and photovoltaic applications. This material is also having interesting photoluminescence properties, and hence finds large number of applications in optoelectronic devices. The structural, optical, acoustical and electronic properties have been correlated with the growth conditions and the nature of substrates. For these reasons, many research groups have shown a great interest in the development and study of this material by various deposition processes such as electro deposition, spray pyrolysis, photo accelerated chemical deposition, microwave heating and chemical bath deposition. The crystalline structure, composition as well as on the major features in the optical and electrical properties of a composite film: PbS–Pb:In. Interest in lead chalcogenide thin films is mainly based on the infrared. In the present work, we reported a dip coating method to produce a thin film of PbS+In [4].

II. EXPERIMENTAL DETAILS

A. Porous Silicon preparation

The cleaned Silicon wafer was etched for 10 minutes at a constant current density of 50 mA cm^{-2} in the HF and ethanol (volume ratio 1:1) solution using single tank cell. The prepared porous silicon (PSi) wafer was rinsed by ethanol and deionized water.

B. Preparing of Lead Sulphide (PbS) solution

Solution –I was prepared by dissolving lead acetate ($Pb(C_2H_3O_2)_2$) in 40 ml methanol(CH_3OH). In solution II, thiourea (CH_4N_2S) was dissolved in 40 ml methanol (CH_3OH). Solution II was added to the solution I drop wise. The mixed solution was stirred, heated at 60°C for about 1 h. After 1 h, the dark chocolate colour powder of PbS is formed. The powder was thoroughly washed several times with methanol and then dried in vacuum. The PbS powder was annealed at 200°C temperature. PbS nanopowder was mixed with 24 ml of m-cresol.

C. Preparing of Indium Sulphide (InS) solution

Indium chloride ($InCl_3$) powder with constant concentration (0.05 M) was dissolved in ethonal and thiourea (0.1 M) solution was added drop by drop into indium chloride solution.

The solutions of InS and PbS were mixed with different volume ratio, such as 1:1, 1:1.5 and 1:2. They are named as PIS1, PIS2 and PIS3 respectively. Each solution was stirred separately and heated at 70°C for 30 mins.

D. Coating Process

The PbInS thin films were deposited on prepared porous silicon (PSi) substrate by dip coating method. Substrates are vertically dipped into the different volume ratio solutions at 60 seconds. The dip coated films (PIS1, PIS2 and PIS3) annealed at 400°C for 30 minutes.

E. Characterization details

Structural properties of the deposited Lead Indium Sulphide thin films were characterised by X'Pert PRO Diffractometer using CuKα1 radiation (λ=1.54060 Å) with operating voltage 40 kV and a current of 30 mA. Surface morphological properties were characterised by HITACHI S-3000 N.

III. RESULTS AND DISCUSSION

A. Structural analysis

From the XRD pattern, it is observed that the intensity of Lead Indium Sulphide peak (7 0 1) is very high compared with other β- In_2S_3 and In_2S_3 peaks. On comparing these XRD patterns the volume ratio affects the intensity of Lead Indium Sulphide thin films. The average crystallite size *(D)* was calculated using well known Debye Scherer formula, [5].

$$D = \frac{0.94\,\lambda}{\beta cos\Theta} \quad \text{nm} \qquad (1)$$

where D is the average crystallite size, λ is the X-ray wavelength (CuKα1 = 1.54060Å), β is the full-width at half-maximum (FWHM) of XRD peaks and θ is the Bragg angle. It is found that the average crystallite size varies as 102.6122 nm, 87.0255 nm and 80.6207 nm with the changes of volume ratio. The lattice parameters of prepared thin film were noted as $a \neq b \neq c$ is a = 38.12; b = 13.80; c = 3.869 and monoclinic system is observed. The films are characterized by a randomly crystallite orientation, similar to the standard JCPDS XRD pattern # 71-0427.

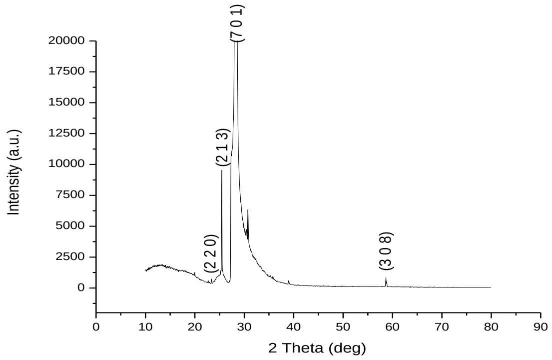

Fig.1.a XRD pattern of PIS1

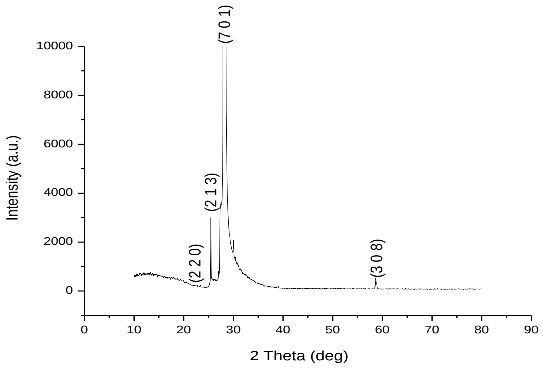

Fig.1.b XRD pattern of PIS2

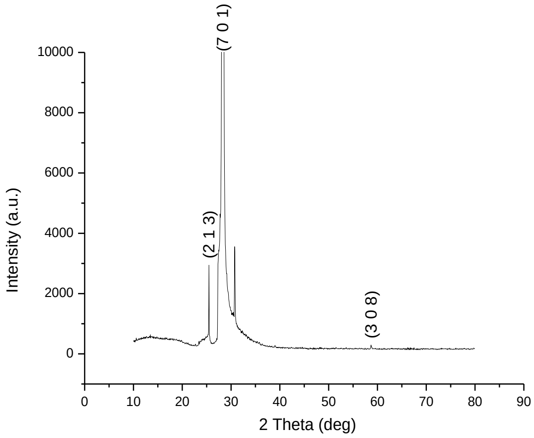

Fig.1.c XRD pattern of PIS3

The dislocation density δ which represents the amount of defects in the film was determined from the formula [6].

$$\delta = \frac{1}{D^2} \qquad (2)$$

Where δ is the dislocation density and D is the average crystallite size of the film.

The calculated value of crystallite size and dislocation density values are tabulated in Table I.

TABLE I.

Sample	Crystallite size (D) nm	Dislocation density δ x 10^{-4}
PIS1	102.6122	0.949734
PIS2	87.0255	1.320404
PIS3	80.6207	1.538533

The observed d-spacing values compared with standard d-spacing values are indicated in Table II.

TABLE II.

Standard d-Spacing (A°)	Observed d-Spacing (A°)			Miller indices (h k l)	c/a	System
	PIS1	PIS2	PIS3			
3.8091	3.8044	3.8022	---	2 2 0	---	Cubic
3.2530	3.25460	3.2571	3.2618	2 1 3	4.2278	Tetragonal
3.1540	3.1572	3.1561	3.1551	7 0 1	0.1015	Monoclinic
1.5737	1.5725	1.5720	1.5720	3 0 8	1.1783	Orthorhombic

B. Surface morphological analysis

Figure 2 shows the SEM micrographs of the surface of PbInS films prepared with different volume ratio of PbS and InS solutions coated on the porous silicon substrate. The PbInS solution was filled into the pores of porous silicon substrate. The particle sizes of the film were found in the range of 102 nm to 80 nm. The XRD and SEM confirmed that the deposited Lead Indium Sulphide films were in nanocrystalline structure.

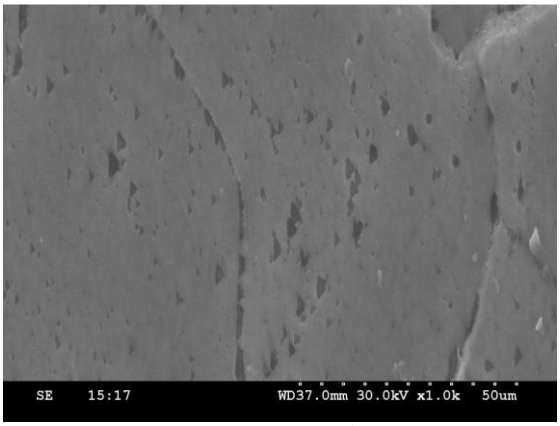

Fig.2. a SEM image of PIS1

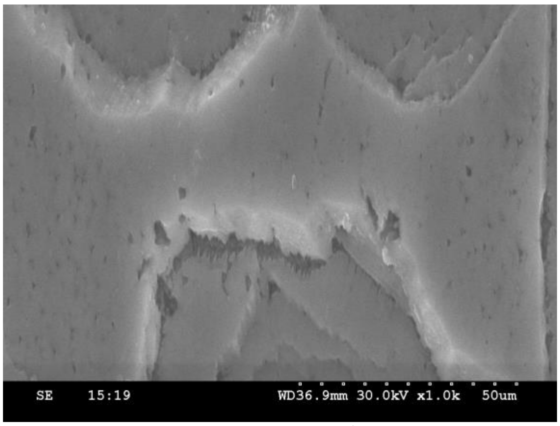

Fig.2. b SEM image of PIS2

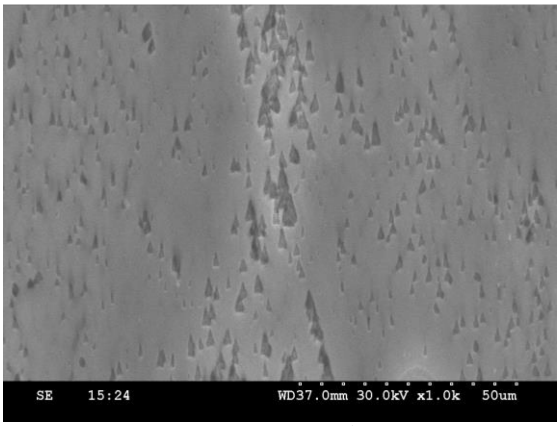

Fig.2. c SEM image of PIS3

IV. CONCLUSION

The PbInS thin films were deposited on prepared porous silicon (PSi) substrate by dip coating method with different volume ratio solutions. The dip coated films (PIS1, PIS2 and PIS3) were annealed at 400°C for 30 minutes. XRD analysis suggested that the deposited film contains PbInS, β- In_2S_3 and In_2S_3 components. The XRD and SEM confirmed that the prepared Lead Indium Sulphide films were in nanocrystalline structure.

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