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Deposited Lead Sulfide Thin Films on Different Substrates with Chemical Spray Pyrolysis Technique

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Abstract: Lead sulfide (PbS) thin films have been deposited onto glass and polymide (PI) substrates at 300 ^{0}C with chemical spray pyrolysis (CSP) technique. Effects of substrate types on the structural properties of the films were studied. Sets of experiments were conducted to optimize the deposition of PbS films with appropriate deposition parameters. To evaluate the melting point temperature of a PI substrate, thermal studies were performed using a Differential scanning calorimeter. The deposited films were analyzed with X-ray diffraction, energy dispersive X-ray and atomic force microscopy to determine their structural properties. DSC measurement confirmed that the PI material has a glass transition temperature (T_g) of approximately 311°C and has no melting peak. X-ray diffraction patterns reveal that the films exhibit the cubic rock salt (NaCl) type structure. PI substrate exhibited the larger roughness than that for the glass because of large particles adsorbed on the PI substrate.

Keywords: Lead Sulfide; Polyimide; Chemical Spray Pyrolysis; Thin Film.

1 Introduction

PbS is a IV-VI compound semiconductor has a cubic lattice with unit cell face center cube [1-3]. It is an important direct and narrow gap semiconductor material with band gap energy of 0.4 eV. PbS thin films have been used in a lot of applications, including electronic and optoelectronic devices [4-6].

Thin films of PbS have been prepared with various physical and chemical thin film deposition techniques, such as chemical bath deposition (CBD) [7], electrodeposition (ED) [8], chemical spray deposition (CSP) [9] and thermal evaporation technique [10]. Among these different techniques, spray technique is advantageous on account of the low-cost and its suitability for forming large area thin films [11].

Research on PbS on flexible polymeric substrates such as polyimide (PI) is gaining immense interests due to their flexibility, light-weight, low-cost (therefore potentially low-cost devices can be fabricated on top of PI), high temperature resistance (typically up to 400°C processing temperature), low coefficient of thermal expansion (CTE), low moisture uptake and high moisture release characteristics, excellent electrical properties and also increased voltage endurance [12]. Due to its superior properties, PI has found its applications as substrates in flexible thin film solar cells, flexible printed circuits and high density interconnects [13,14].

In this work, an attempt has been made to study the structural properties of the PbS thin films grown by chemical spray pyrolysis technique on glass and polymide substrates. This paper aims to use of PI plastic as a substrate for thin-film solar cells with chemical spray pyrolysis technique.

2 Experimental Detail

2.1 Substrates cleaning

In this experiment, glass and PI plastic were used as substrates. The glass and PI plastic substrate was cleaned by alcohol for 10 min to remove contamination. After the cleaning process, all of the substrates were rinsed with distilled water (DI water). The samples were then dried with nitrogen (N_2) gas.

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2.2 Materials

Lead (II) acetate trihydrate ($Pb(CH_3CO_2)_2 \cdot 3H_2O$; 99.999%), Thiourea ($CS(NH_2)_2$), ≥99.0%) were acquired from Acros Organics.

2.3 Preparation of PbS Solutions

A typical PbS solution procedure involves:

1. Thiourea solution [$CS(NH_2)_2$]: This solution was prepared with molarities (0.1M), from solving (0.761gm) of thiourea in (100ml) of distilled water and was marked **Solution 1**. This solution was vigorously stirred using a magnetic stirrer about 10 minutes.

2. Lead Nitrate solution [$Pb(CH_3CO_2)_2 \cdot 3H_2O$] : This solution was prepared with molarities (0.1M), from solving (2.78gm) of lead acetate in (100ml) of distilled water and was marked **Solution 2**.

This solution was vigorously stirred using a magnetic stirrer about 10 minutes. A Solution 1 was added to Solution 2 and vigorously stirred using a magnetic stirrer and was marked **Solution 3**.

PbS thin films were deposited form a solution 3 by chemical spray pyrolysis technique on glass and PI substrates. The description of experimental set-up of (CSP) system is presented in **Fig. 1**. In order to get uniform thin films, the height of the spraying nozzle and the rate of spray process were kept constant during the deposition process at 35 cm and 15 cm^3/min. The spraying process lasted about 6 second. The period between spraying processes was about 1 min; this period was enough to avoid excessive cooling of the substrate. PbS thin film was deposited on glass and PI substrates at a temperature of 300 ^{0}C and a concentration of 0.1 M. The crystallographic structure of the PbS thin films deposited on the glass and PI substrates was determined with a high resolution X-ray diffractometer system (Model: Panalytical Empyrean) with CuKα radiation (λ) of 0.154 nm. The compositions of the PbS thin films were estimated with energy dispersive X-ray analysis (EDX) (Model JSM—6460 LV).The surface morphology of PbS thin films was determined with atomic force microscope (AFM, Model: Ultra Objective). The thicknesses of the PbS on the glass and PI substrates are of the order of 300 nm, which were measured with an optical reflectometer (Model: Filmetrics F20)

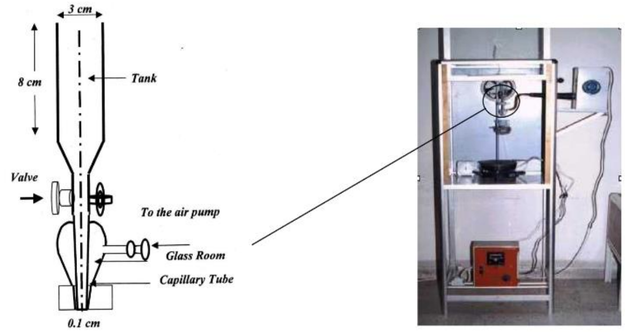


Fig.1. Experimental Set-up of Spraying Apparatus (**right**), and Layout of enlarged spraying glass nozzle (**left**).

3 Results and discussion

Fig. 2 plots the second DSC heating curve of the PI substrate heated from 30°C to 500°C in N_2 ambient (heating rate = 20°C/min). The upwards arrow indicates endothermic reaction. From the figure, glass transition temperature (T_g) of approximately 311.95°C is evident (as shown by the inflection point). This transition is indicated by an increase in heat capacity due to increased molecular motions in the PI substrate [15].

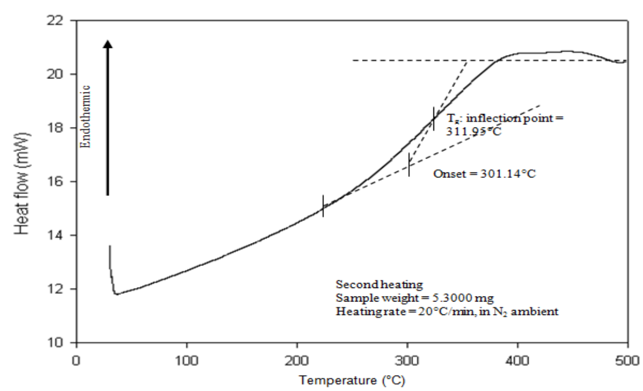
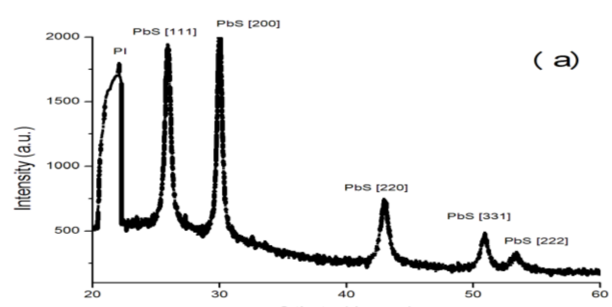


Fig. 2 DSC plot of PI substrate.

No melting peak is observed within this temperature range (30 – 500°C) which conforms to the intrinsic property of standard Kapton polyimide 300HN that possesses no melting point [16].



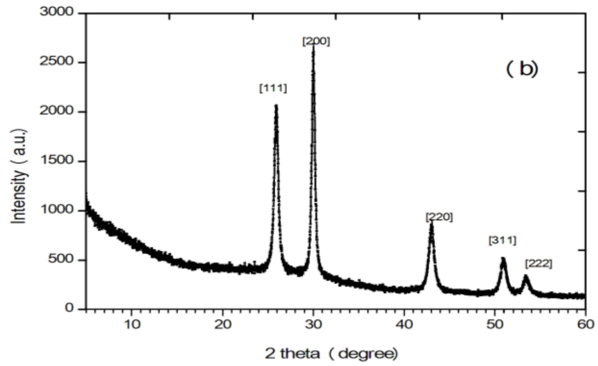


Fig.3 XRD patterns of PbS thin film on **a.**PI and **b.**glass substrates.

The X-ray diffraction patterns of PbS film on PI and glass substrates are shown in **Fig. 3** PbS film have cubic rock salt (NaCl) type structures. XRD patterns of the PbS thin films showed sharp [1 1 1] and [2 0 0] peaks along with minor peaks of [2 2 0], [3 1 1] and [2 2 2] planes to cubic structure of PbS thin films, as confirmed by standard ASTM card (No. 030660020).For the PbS thin films on the PET substrate, the main peak, which corresponds to the PI substrate, was observed at the angle $2\theta = 22.08^0$ and has a high intensity, as seen in **Fig. 3.a.**This behaviour is in agreement with that reported in the literature [17].

Fig.4 shows the EDX results for the PbS thin film deposited on glass and PI substrate. The EDX analysis confirmed the composition of lead and sulfur in the PbS film. The presence of C and O peaks was due to the PI substrate in **Fig. 4.b**. The presence of Si and O peaks in **Fig. 4.a** is due to the glass substrate

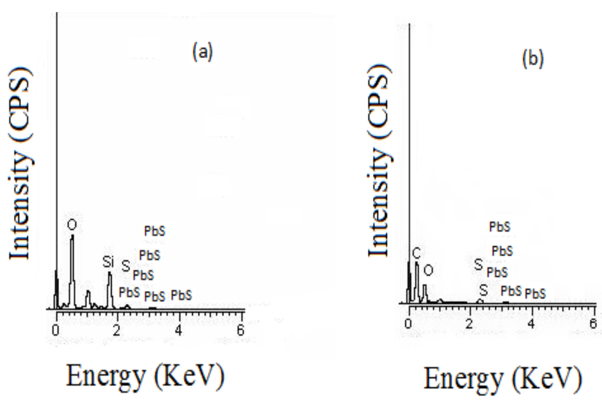


Fig. 4 EDX results for PbS thin films deposited on **a.** glass substrates and **b.** PI substrates

AFM images of the surface morphologies of PbS thin films onto glass and PI substrates are shown in **Fig. 5.** The root mean square (RMS) for PI thin films on glass is **9.62** nm, and that the RMS for PI substrates is **12.62** nm. The increase of roughness is due to the large particles that are absorbed on the PI substrates. Obviously, the PbS films on glass have smoother surfaces than those on PI substrates.

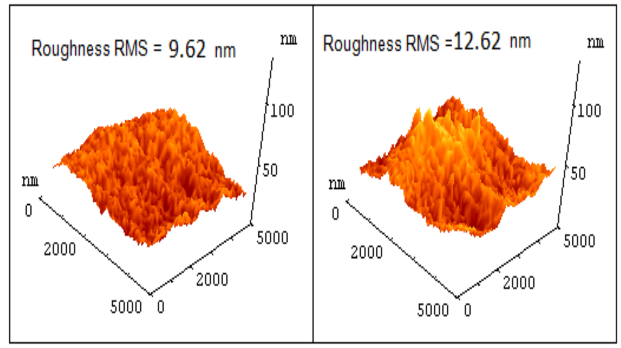


Fig. 5 AFM analysis of PbS thin films deposited on **a.** glass substrate, **b.** PI substrate.

4 Conclusions

Lead sulfide thin films were deposited onto polymide and glass substrates at 300 ^{0}C with chemical spray pyrolysis technique. The PbS thin films were found to have different roughness values when deposited on glass and compared to when they are deposited on PI substrates. X-ray diffraction patterns confirm the proper phase formation of the PbS. DSC measurement confirmed that the PI material has a T_g of approximately 311°C and has no melting peak.

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