# Effect of changing substrate temperature of PbI<sub>2</sub> thin films deposited by spray technique on the structural ,optical and electrical properties

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## Abstract

Polycrystalline layers of Lead iodide have been prepared using a spray technique on glass substrates for potential application to nuclear detection .This paper studied The influence of changing substrate temperature of  $PbI_2$  films on the structural, optical and electrical properties has been investigated at Different substrate temperature  $(150.200.250)^{\circ}$  C.

#### **1-Introduction**

The development of compound semiconductors intended for room temperature radiation detection has been performed for several decades [1]. Lead iodide PbI<sub>2</sub> nuclear radiation detectors are excellent candidates for many application which require portability and low price. They are suitable to be used for high out door temperature as detector for X and v ray radiation .High atomic number materials such as lead iodide and mercuric iodide are very sensitive to gamma and x-ray, compard to low atomic number materials such as silicon and germanium [2].  $PbI_2$  has convenient physical properties for this task, like atomic number of its elements (Pb) =82, I=53, density (6.2 g/cm<sup>3</sup>), mass absorption coefficient (only 7  $\mu$ m of lead iodide are sufficient for 90% absorption of 6 keV radiation and 1560µm for 120 k eV radiation), band gap and phase diagram. Due to the wide band gap (2.3-2.5) eV, detectors of this material would be able to operate at room temperature and even above [1]. Pbl<sub>2</sub> have other properties like lower vapor pressure, thermal stability and relative environments' stability [3]. There are many techniques to synthesize  $pbI_2$  thin films,

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including gel method [4], thermally evaporation, physical vapor deposition, a solution deposition[5], and spray pyrolysis[6]. Among available techniques spray pyrolysis are simplest and relatively low cost once that is dose not require expensive equipments [7], and because it can be easily expanded for large areas .

#### 2- Experimental

Polycrystalline PbI<sub>2</sub> thin film samples were prepared on glass substrates using spray technique. The production of thin films from the vaporization of a solution [9]. Advantages of this method are simplicity and relatively low cost once that is dose not require expensive equipments and because it can be easily expanded for large areas and deposition time about 2.5 hour was adopted[10]. Lead iodide solution is prepared from lead iodide powder (0. 2g) dissolved in distilled water of 100 ml at  $(100^{\circ}\text{C})$ with 0.02 PVA, then the solution was heated with continuous mixing until the yellow powder is completely dissolved. The solubility of Pbl<sub>2</sub> increases in hot water. The starting material was lead iodide powder from (England) with purity of 99.99%. The main residual impurity in the base material was, PVA(BDH chemical Ltd ) which is used as binding material and added in the initial solution prior to the solution heating. The  $pbI_2$  thin film were deposited by hand made spray technique shown in figure(1). The solutions are sprayed on the heated substrates at various temperatures (150,200, 250)  $C^{\circ}$ , the time of the deposition was (4sec) spray and (26 sec) to stop each so mointul temperature ( $\pm$  5° C).



Figure (1): The set up of the spray technique system.

## **3-Results and Discussions**

#### Structural Properties 3-1X-ray Diffraction

Through studying the X-ray diffraction powder, one can understand the polycrystalline growth nature of PbI<sub>2</sub> thin films prepared by spray technique deposition on glass substrates at 150, 200 and 250° C.

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## **Effect of Substrate the Temperature**

From the X-ray diffrograms the planes orientation were determined; grain size and micro-**Strain** ( $\sigma_s$ ) of PbI<sub>2</sub> thin films were calculated. Figure(1A,B,C) show the XRD of samples prepared under different substrate temperatures. Peaks are observed at ( $2\Theta$ = 12.547°, 25.302°, 8.547° and 52.255°) correspond to (001),(002),(003) and (004) planes respectively, this results are in a good agreement with results achieved by authors [11,12] The inter plane spacing is calculated by using diffraction Bragg equation [6]:

Where

 $\Theta$ : is a angel of diffractions

 $\lambda$  :is the wavelength of radiation  $\,CuK\alpha.$ 

n :is a positive integer.

d :is the inter-planar spacing.

The average grain size is deduced by using Scherrer equation

 $G.S=0.9 \lambda/\Delta \cos\Theta \dots (2)$ 

 $\lambda$ : is the wavelength of radiation CuK $\alpha$ 

The micro-Strain is caused during the growth of the film, and will be raised from stretching or compression in the lattice. This Strain can be calculated from the relation [6]:

$$\sigma_s = \left[\frac{d_{JCPDS} - d_{XRD}}{d_{JCPDS}}\right] \times 100\% \dots (3)$$

Where

d <sub>JCPDS</sub> : is the inter-planar spacing standard for card JCPDS.

 $D_{XRD}$ : is the inter-planar spacing for examination XRD. $\Delta$ : the full width half max (FWHM)

Table (1) summarize the obtained values of  $2\theta$ , d, average grain size and micro-Strain for undoped films. Despite a visibly good crystallinty, many reflections are missing from their diffraction patterns, as shown in Figure (1A,B,C). From this table the greatest grain size are those of layers deposited in temperature 200° C.

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Figure (2) XRD patterns for  $PbI_2$  at different substrate temperature and 1µm thickness. a)150° C b)200° C c)250° C

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Table (1): Analysis of the XRD study of undoped PbI <sub>2</sub> films						
Sample	20	d	(hkl)	Grain size	σ <sub>s</sub>	
	(degree)	( <b>A</b> <sup>0</sup> )		( <b>nm</b> )	%	
Sample	12.500	7.073	(001)	29.41	1.332	
(1) at	25.5079	3.49	(002)	30.9	0.029	
° C150	38.5427	2.3355	(003)	31.7	0.344	
Sample	12.732	6.9472	(001)	29.4	0.516	
(2) at	25.4861	3.49217	(002)	44.8	0.029	
° C200	38.6385	2.32838	(003)	29.19	0.344	
Sample	10	6.9552	(001)	29.395	0.988	
(3) at						
° C250						

Figure (3) shows the variation of micro-**Strain** ( $\sigma_s$ ) as a function of deposition temperature This Figure shows that the less micro-**Strain** ( $\sigma_s$ ) value is at 200C°, which means that the 200C° is the optimum temperature and this is due to that the lattice constant at this temperature is close to the lattice constant of card standard JPCD.



Figure (3) shows the variation  $\Pi \ \Box C$  train ( $\sigma_s$ ) with deposition temperature.

The plots of the variation  $2\Theta$  as a function of Temperature is shown in figure(4), and it is noticed that the 200c° is the most best temperature. When these results compared with the JCPD the  $2\Theta$  (12.732) are the most matched.



Figure (4) shows the variation of  $2\theta$  as a function of substrate temperature.

## **3-2Scanning Electron Microscope (SEM)Analysis**

By using scanning electron microscope SEM, it can be observed the micrographs for films deposited at (150,200 and 250 °C).  $PbI_2$  forms 'hexagonal', tilted image as shown in Figure (5),(6),(7)and found small white zone over spherical grain shape ,it present PVA poly venal alcohol binding material. For higher substrate temperature at 250 °C not found PVA in the image because evaporated. This result is in a well agreement with literature [13],[14].



Figure (5) SEM result for sample at 150° C



Figure (6) SEM result for sample at 200° c.

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Figure (7) SEM result for sample at 250° c

## **3-3-Optical Properties of PbI<sub>2</sub>**

optical properties of the samples prepared by spray technical deposition have been investigated. These properties include the followings:-

## 3-3-1Transmission

Figure (8) shows the spectral optical transmittance in the wavelength range (400-1100 nm) for film deposited at different substrate temperatures,. In this figure a sharp increment starts around (~520 nm). This result is in good agreement withresult of refference [16]. The highest value of the transmittance was at 200°C and this means that the film with high homogeneity.



Figure (8): Optical transmission spectrum for Pbl<sub>2</sub> films

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#### **3-3-2Absorbance**

Figures (9) show the variation of absorbance as a function of wavelength, at wavelength range (400-1100 nm) for sample at different substrate temperature .It is shown that there was increasing in the absorbance with the increasing of substrate temperature, and the absorbance edge is unchanged with the change of substrate temperature .



Figure (9): Absorbance vs. wavelength for sample for different substrate temperatures. **3-3-3 Optical Transitions** 

To understand the optical and electrical properties of the prepared films, it is very necessary to study and estimate the mean value of the optical band gap. This value depends on the films structure, the arrangement and distribution of atoms in the crystal lattice.

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The spectral absorption coefficient curves indicate the direct transitions. The optical band gaps were determined from the plots of  $(\alpha hv)^2$  versus (hv) where the extrapolation of the straight line region gives the optical band gap.

In Pure samples are deposited at the different substrate temperatures is show in Figure(10). The band gap values of samples are increase with increasing substrate temperature and these values are in a good agreement with publish work .[15]



Figure (10) The energy gap of sample deposited at 150,200 and 250  $^{\circ}$  C  $$h \upsilon$  (ev)

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#### **3-4The Electrical Properties of PbI**<sub>2</sub>

The electrical properties of the pure  $PbI_2$  and doped  $PbI_2$  thin films deposited on glass substrate by spray technique at substrate temperature 200 °C are investigated .

#### **3-4-1 Resistivity**

From the I-V curve the resistance  $(R_{es})$  calculated, and from the inter electrodes distance (l) and the contact area (A) the resistivity ( $\rho$ ) was calculate [6]:

And the area calculates by [6]:

$$A = B \times d$$
 ------ (4)

Where (B) is the width of the electrode and (d) is the layer thickness.

#### Substrate temperature effect

Figure (11) show the relation between resistivity and substrate temperature, it was found that there is decrease in resistivity with increasing substrate temperature, this could be attributed to the evolution of the adsorbed oxygen at film surface and grain boundaries which allow the increase of electrons and decrease in the potential barrier at grain boundaries. These results are consistence with other published results such as the results of



Figure (11): The electrical resistivity as a function of different substrate temperatures  $150,200,250^{\circ}$  C.

#### 4-Conclusion :

The XRD shows polycrystalline structure, with four main peaks: (001), (002), (003), and (004).the demented peak (001). The optimum temperature 200C° PbI<sub>2</sub> thin film when compared with the JCPD the  $2\Theta$  (12.732) are the most match. Less strain of the thin films were deposited at 200° C. The SEM result found PbI<sub>2</sub> forms 'hexagonal' with spherical grain shape and found small white zones over spherical grain shape ,may be it present PVA poly venal alcohol binding material. the value of band gap varying (2.19 -2.4) eV also it was found that the energy gap for PbI<sub>2</sub> thin films are increase with increasing substrate temperature, the high resistivity (2.67x10<sup>7</sup> $\Omega$ .cm) for sample deposited in 150° C.

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الخلاصه

تم تحضير نماذج من يوديد الرصاص المتعدد التبلور باستخدام تقنية الرش بالترسيب على قواعد زجاجية تستخدم في التطبيقات النوويه في هذا البحث درس تأثير تغير درجة حرارة الركيزة لاغشية يوديد الرصاص على الخصائص التركيبية ،البصريه والكهربائيه للنماذج المحضرة عند درجات حرارة مختلفة (200,150، 250) درجة مئويه .