Constructing Spray Pyrolysis System For The Preparation of Thin Films and Study The Optical Properties of PbO Films

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ABSTRACT In this paper, the design of spray pyrolysis system to prepare thin films of various materials system. The study showed that films prepared with good characteristics. films were prepared from lead oxide PbO this way. The study of the optical properties of these films and through recording the absorbance spectrum and transmittance spectrum of films prepared in the wavelength range (300-1100) nm . Optical constant for thin films prepared such as absorption coefficient, the extinction coefficient , the refractive index and the optical energy gap was calculated as well. films prepared found that it has a direct energy gap value 3.1eV.

1. INTRODUCTION

Among different chemical methods, spray pyrolysis technical is most popular today because its application to produce a variety of conducting and semiconducting materials [1]. The fundamental principle involved in the spray pyrolysis technical is the thermal decomposition of the compound to be deposited salts. Spray pyrolysis technique has a list of advantages. Spray pyrolysis technical is a simple and low cost to set up semiconductor thin films. Has the capacity to produce wide area, high quality films attached to standardized thickness [2]. Transparent conducting oxide (TCO) thin films such as PbO, ZnO, SnO₂, In₂O₃ and MoO₃ have been studied in detail by many researchers [3,4]. These TCO_s find extensive applications in thin film transistors, solar cells, phototransistors, optical storage devices, gas sensors, photo-thermal and photovoltaic conversions [5-8]. PbO exists in tetragonal (α -phase) and orthorhombic (β -phase) structures at low and high temperatures respectively [9]. The difficulty of preparing exclusively single phase α -or β -PbO was pointed out earlier. The α -PbO was obtained earlier by pulsed laser ablation and spray pyrolysis [10]. The α -PbO was transformed to the meta-stable β -PbO when heat treated beyond 489 °C [11].

2. EXPERIMENTAL DETAILS

2.1 First: spray pyrolysis system

A simple homemade spray pyrolysis experimental setup was employed to prepare thin films, The system consists of several parts have been arranged so as to make use of them in the preparation of various the films on various substrates. Figure (1) shows the system in all its parts, and the system consists of the following devices:

Fig. (1): Chemical Spray Pyrolysis System.

2.1.1 Spray nozzle

It is made up of plastic with an iron head diameter 0.1mm contains within it on a piece of iron and other passes through the air and related tube two pieces, one for the transfer of compressed air and the second relates to a small receptacle containing solution from the material to be deposited. As shown in Figure (2).

Fig.(2): Spray nozzle

2.1.2 Hot plate

The iron disc (with diameter 16 cm and thickness 0.7 cm to which 2000 W heating coil is fixed) served as a hot plate. A maximum temperature of 700 ± 20 °C can be achieved with this arrangement. The chromel-alumel thermocouple is used to measure the temperature of the substrates and is fixed at the centre of the front side of the iron plate. The temperature of the hot plate is monitored with the help of temperature controller, as showing in Figure (3).

Fig.(3): Hot plate

2.1.3 Air pump

It has been used pneumatic pump compressed air to drive inside the tube connected to the spraying. The air pressure is stable here. The compressed air is the useful pushing particulate material deposited is that we want to spray toward the substrate. The air pump show in Figure (4).

Fig.(4) :Air pump

2.1.4 Electronic Temporary

Through its control the number of sprinkles and the time of spraying through a pump or stop pump the air. Where we need to pause after each spray solution on the hot substrate to regain substrate temperature. Because after all spraying the substrate will collide. The electronic temporary show in Figure (5).

Fig.(5) :Electronic Temporary

2.2 Second: Prepared Thin Films

2.2.1 Solution Preparation (PbO)

The PbO films were prepared by using an aqueous solution of Lead Acetate Dihydrate $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ with concentration 0.1 M dissolved in 100 ml of distilled water. The mixture was stirred by (Magnetic stirrer) at 40 °C for 25 min and then it was allowed to cool to the room temperature with continuous stirring.

$\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ is a powder material which has a white color and its molecular weight (379.33 gm/mol). For calculating mass of $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ in the current experiment the following equation was used:

$$M = \frac{W_t}{M_{wt.} \times \frac{V}{1000}} \quad (1)$$

Where:

M: concentration of molarities.

W_t: weight of $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$

M_{wt.}: molecular weight of $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$.

V : volume of distilled water, (100 ml).

2.2.2 Preparation (PbO) thin film

A simple homemade spray pyrolysis experimental setup was employed to prepare (PbO) thin films on glass substrates ($35 \times 25 \times 1.35 \text{ mm}^3$) at a substrate temperature of 375 °C. The deposition parameters such as spray nozzle-substrate distance (30 cm), spray time (5 s) and the spray interval (1 min) were kept constant. The carrier gas (filtered compressed air) flow rate was maintained at 6 l/min at a pressure of $6.5 \times 10^4 \text{ Nm}^{-2}$.

Optical transmittance spectra in the wavelength ranging 300–1100 nm were recorded using UV Visible spectrometer (Shimadzu, UV-1601).

3. RESULTS AND DISCUSSION

3.1 Thickness of the (PbO) films

It was estimated from the mass of the deposited materials using the gravimetric method. Surface diffusion (on the substrate) of the sprayed chemical species makes it very complicated to form a step (in pyrolytic deposition process) required by a conventional thickness profilometry. The standard density of PbO (9.35 g/cm^3) [12] was used for calculation. The thickness of the films found 150 nm.

3.2 Optical properties

Figure (6) show the absorption spectra of the PbO thin film, The average absorbance for the wavelength of visible region (500–850 nm) varies between 14–12 %.

Fig.(6): Absorbance spectrum as a function of wavelength for PbO film.

Figure (7) shows the transmittance spectra of the PbO thin film, The average transmittance in the wavelength of visible region (500–850 nm) varies between 72–75 %.

Fig.(7): Transmittance spectrum as a function of wavelength for PbO film.

Figure (8) shows the absorption coefficient (α) for the prepared thin film which calculated from eq. (2). The values of α for thin film are found to be greater than 10^4 cm^{-1} in the visible region, which means that the films have a direct optical energy gap [13], so that the value of r in eq.(3) is equal to $\frac{1}{2}$.

$$\alpha = 2.303 \frac{A}{t} \dots\dots\dots (2)$$

Where :

A : absorption.

t : thickness of thin film.

Fig.(8): Absorption Coefficients as a function of wavelength for PbO film.

The direct optical energy gap (E_g) was calculated by using the relation (3) with $r = \frac{1}{2}$, and from the Figure (9), the values of E_g were determined from the intersection point of the extrapolation of a linear curve with $h\nu$ axis. The energy gap equal 3.1 eV.

$$\alpha h\nu = B (h\nu - E_g)^r \dots\dots\dots (3)$$

Where:

E_g : energy gap between direct transition.

B: constant depended on type of material.

r : exponential constant, its value depended on type of transition, $r = \frac{1}{2}$ for the allowed direct transition.

$h\nu$: photon energy.

The values of extinction coefficient (K) are calculated using the relation (4). The K value is plotted vs. λ .

$$K = \alpha \lambda / 4\pi \quad (4)$$

Where λ : is the wavelength of incident photon rays.

Fig.(9) $(\alpha h\nu)^2$ as a function of $h\nu$ for PbO film.

The minimum value of K equal to 56×10^{-3} at wave length 400nm and increase with increasing λ to 105×10^{-3} at $\lambda = 1100$ nm as show in Figure (10). This means that there are different absorption mechanisms, depending on the film structure.

Fig.(10): Extinction coefficient as a function of wavelength for PbO film.

The refractive index is the ratio between the speed of light in vacuum to it's speed in material which doesn't absorb this light . So the value of n begins when K is constant [1]. The refractive index (n) was calculated from relation (5). The values of n vs. λ were shown in Figure (11).

Fig.(10): Refractive index as a function of wavelength for PbO film.

From the graph n value decreasing with increasing λ , the explanation of this behavior may be related to the polarization of thin film because n depends on material polarization where with increasing polarization the velocity of light was decreased so n changed. The polarization depends on crystalline and on grain size of thin film so these depend on preparation conditions.

$$n = \left[\left(\frac{1+R}{1-R} \right)^2 - (K^2 + 1) \right]^{1/2} + \frac{1+R}{1-R} \quad (5)$$

Where :

R : The reflectance can be calculated from the following equation

$$R + T + A = 1 \quad (6)$$

4. CONCLUSIONS

Through the results of a fortress by the optical to geared for films PbO and compatibility with research and studies numerous conclude that the system that was built for the preparation of thin film materials solid technology, chemical spray pyrolysis useful and accredited for the preparation of thin films certified results for the purposes of research, studies and valid for use in multiple applications results.

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