



Optical properties of amorphous and crystalline tris(8-hydroxyquinoline) indium films



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ABSTRACT

Tris(8-hydroxyquinoline) indium (Inq₃) films were physically deposited via thermal evaporation technique and were subsequently thermally annealed at different temperatures in air atmosphere. The effect of annealing on the structural and optical properties of the films was investigated. X-ray diffraction showed that the as-deposited and annealed films at 100 and 200 °C have amorphous structure, whereas the annealed films at 270 and 300 °C have crystalline structure in which a preferred orientation of growth is obtained. The evolution of the optical properties of the films due to annealing has been correlated with their structural properties. The optical properties were studied in terms of spectrophotometric measurements of the transmittance and reflectance over the spectral range 200–2500 nm, from which the refractive and absorption indices were calculated using Murmann's exact equations. The single oscillator and Drude models were applied for determining the dispersion parameters of the films. Analysis of the absorption coefficient revealed that the films have direct allowed optical band gap of 2.77 eV, which is increased to 2.85 eV as a result of annealing. The thermal properties were studied by means of differential scanning calorimetry and thermogravimetric measurements.

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1. Introduction

Organic semiconductors with promising optical and electrical properties are recognized as an important class of interesting materials for applications in advanced electronic and optoelectronic devices in which their physical properties can be tuned by chemical modification on the molecular frame. The domain of organic materials continues to be of great scientific and commercial interest due to other advantages such as low cost and ease of processing. They are currently used as active layers in large number of advanced electronic and optoelectronic devices such as light emitting diodes [1], thin film transistors [2] and solar cells [3], as alternatives to expensive inorganic materials.

Tris(8-hydroxyquinoline) aluminum (Alq₃) is the first efficient material in organic light emitting diode (OLED) devices, which was reported in 1987 by Tang and Vanslyke [4]. Since then, intense efforts have been dedicated to develop and improve the efficiency of such diodes. Most of the recent studies have been directed towards improving morphological stability [5], optimizing the device

characteristics [6], understanding the charge carrier transport mechanisms [7] and tuning the emission spectrum of OLED [8]. Besides, Alq₃ is used as a buffer electron transport layer [9] and dopant material in organic solar cells [10]. Ever since the success of Alq₃ in these devices, a lot of attention has been paid to synthesize and investigate other counterparts. In this regard, Inq₃ has been introduced as promising candidate in OLEDs. Triggered by its interesting properties, efforts have been paid for further material analysis and characterization. The study of the optical behavior of such materials in thin film form is a prerequisite for the successful device fabrication. Shukla and Kumar [11] have been investigated the optical properties of Inq₃ and Alq₃ films by using spectroscopic ellipsometry. The study showed that the refractive index of Inq₃ films is higher than Alq₃ films. Thangaraju et al. [12] showed that the Inq₃ films have less photoluminescence intensity compared with that of Alq₃ thin film. Thermal annealing was used as an effective tool for influencing the structural and optical properties of Alq₃ [13–15] and Gaq₃ [16] films.

To the best of my knowledge, the effect of thermal annealing on the structural and optical properties of Inq₃ thin films has not been extensively reported. The target of this work is to study the influence of thermal annealing temperature on the structural properties

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and optical functions of thermally evaporated Inq_3 films over the spectral range 200–2500 nm. Thermal annealing is a common used process in optical applications, which is helpful tool to improve the film properties. For example, annealing has been used as a vital process to improve the electrical conversion efficiency of solar cells. Thus, it is important to find the optimum annealing temperature, at which the film could possess better optical properties.

2. Experimental

2.1. Synthesis

Tris(8-hydroxyquinoline) indium was prepared according the procedure given elsewhere [17]. In typical synthesis, 0.5 g of 8-hydroxyquinoline was dissolved in a mixture of 10 ml double distilled water and 2.5 ml of glacial acetic acid. The mixture was vigorously stirred until an orange transparent solution was obtained. Then, 0.3 g of indium nitrate hydrate was dissolved in 20 ml of double distilled water and stirred until a clear solution was obtained. The two solutions were mixed together and stirred for 5 min. After that, ammonium hydroxide was added to the mixture solutions, drop by drop with continuous stirring to get maximum precipitation. A yellow-green precipitate was filtered out and washed 15 times with double distilled water and dried at 80 °C under argon gas flow.

2.2. Films deposition and measurements

Inq_3 films were deposited onto pre-cleaned glass and optically flat fused quartz substrates using thermal evaporation technique under a base pressure of about 10^{-4} Pa by using high vacuum coating unit (Edwards, E 306 A). Initially, Inq_3 powder was placed inside a quartz crucible that has been surrounded by a tungsten coil capable of supplying sufficient heat to the materials upon passing an electrical current through this coil. Current of about 40 A was enough to start sublimating Inq_3 powder. The rate of deposition and film thickness were controlled during the evaporation by using a quartz crystal thickness monitor. The rate of deposition was adjusted at 0.15 nm/s. The film thickness was determined as 386 nm after deposition by spectrophotometric method. The substrates were kept at room temperature (25 °C) during the deposition process. Prior to deposition, the substrates were cleaned ultrasonically for 15 min with hot distilled water, followed by 10 min rinsing in an ultrasonic bath in acetone, ethanol and distilled water, respectively. Finally, the substrates were dried thoroughly under argon gas. Glass substrates were used for structural analysis, whereas quartz substrates were used for optical measurements.

Inq_3 films were thermally annealed at temperatures of 100, 200, 270 and 300 °C in air atmosphere. Annealing was performed in dark condition and the time of annealing was two hours. The temperature of the samples was recorded by using NiCr–NiAl thermocouple with an accuracy of ± 1 °C.

The X-ray diffractometer (Philips X' pert) with Ni-filtered CuK_α radiation was used to identify the structural characteristics of both powder and films. The measurements were performed in the diffraction angle (2θ) range 4–80° with a step size of 0.02 (2θ /s). Morphology of the films was studied by field emission scanning electron microscope (Quanta FEG 250).

The optical transmittance and reflectance spectra of the films were recorded over the wavelength range 200–2500 nm using double beam spectrophotometer (JASCO V-570 UV–Vis–NIR). The measurements were performed under ambient conditions. The absolute values of total measured transmittance, T_m , after introducing corrections resulting from the absorbance and reflectance of

the substrate were calculated by Ref. [18]:

$$T = T_m(1 - R_q) \quad (1)$$

where R_q is the reflectance of reference quartz substrate. The spectrophotometer was also provided with a reflection attachment to measure the reflectance. The absolute values of the total measured reflectance, R_m , after introducing corrections resulting from reflectance of the substrate and reference mirror were calculated by:

$$R = R_m R_{Al} \left\{ 1 + (1 - R_q)^2 \right\} - T^2 R_q \quad (2)$$

where R_{Al} is the reflectance of the reference aluminum mirror.

The thermogravimetric (TG) and differential scanning calorimetry (DSC) measurements were performed at heating rate of 10 °C/min and in nitrogen environment by thermogravimetric analyzer SDTQ 600 TA.

3. Results and discussion

3.1. Structural properties

Fig. 1 depicts the X-ray diffraction patterns of Inq_3 in both powder and thin film conditions. It can be observed that the spectrum of the powder has different peaks with different intensities implying polycrystalline structure. Upon thermal deposition, no identified diffraction peaks are found which give the ambiguous evidence of amorphous structure. The same behavior can also be seen for annealed films at 100 and 200 °C. Upon rising annealing temperature up to 270 °C, a preferred orientation of growth along the diffraction peak centered at diffraction angle of 7.03° is obtained indicating crystalline structure. The intensity of this peak is slightly increased by annealing at 300 °C indicating slight improvement of films crystallinity. The appearance of this diffraction peak at high annealing temperatures is attributed to the sufficient increase in supply of thermal energy for crystallization, re-crystallization and growth of grains in the films. As-deposited Alq_3 [13] and GaQ_3 [16] films are also found to grow with amorphous structure. Thermal annealing at 200 °C is found to crystallize Alq_3 films and thermal annealing at 255 °C is found to crystallize

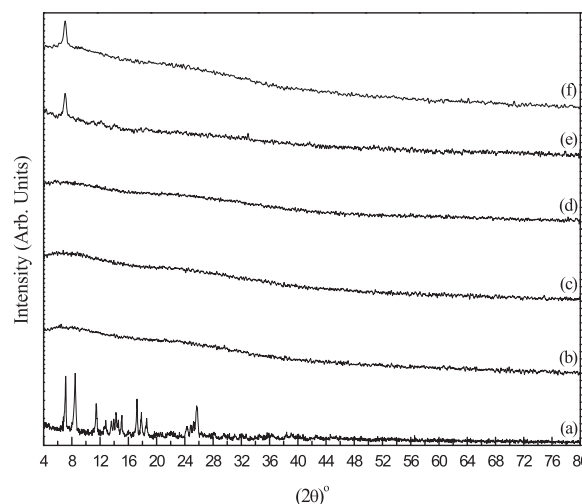


Fig. 1. X-ray diffraction spectra of Inq_3 : (a) powder, (b) as-deposited film, (c) annealed films at 100 °C, (d) annealed films at 200 °C, (e) annealed film at 270 °C and (f) annealed film at 300 °C.

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