

Structural and thermal characteristics of the fast-deposited parylene substrate for ultra-thin organic light emitting diodes



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ABSTRACT

Structural and thermal characteristics of fast-deposited poly-*para*-xylylene (parylene) films were investigated. A transparent parylene film exhibiting high transmittance and low haze was successfully deposited with an evaporation speed of >500 nm/min. Based on Fourier transform infrared spectroscopy and X-ray diffraction, it was demonstrated that the fast-deposited parylene films possessed the same molecular structure and d-spacing as slow-deposited parylene films. However, differential scanning calorimetry confirmed that the fast-deposited parylene films exhibited lower melting temperatures and melting enthalpies than the slow-deposited parylene films. These results imply that the fast-deposited parylene films may experience restricted movement of monomers on the cold substrate during polymerization in terms of the product film. Ultrathin flexible OLEDs with a green fluorescent emitter were also successfully fabricated on this fast-deposited parylene substrate.

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1. Introduction

Poly-*para*-xylylene (parylene) and substituted parylene have been used in the conformal coating industry to protect electrical devices and mechanical parts from outer stresses, such as chemical reactions or physical stress. Compared to conventional polymer films prepared according to solution processes, these parylene films can be polymerized on a surface directly from the gas phase under the vacuum condition [1]. The resulting pinhole-free parylene films have a number of advantages, including high chemical and electrical resistance, high transparency, and low gas permeability, among others. Recently, parylene derivatives such as parylene D (poly-dichloro-*para*-xylylene), parylene C (poly-chloro-*para*-xylylene), and parylene F (poly-tetrafluoro-*para*-xylylene) have been widely utilized in micro-electromechanical sensors, flexible electronics, and in the medical industry [2,3]. In the area of flexible electronics, wide-ranging applications such implementation in encapsulation layers (OLED, OPV), flexible substrates (sensor, OLED), interlayers for imprinting processes, insulating layers (OTFT), and planarization layers (OLED) have been investigated [1–8].

The chemical vapor deposition of parylene was introduced by

Gorham and consists of four steps, which take place in an evaporation chamber, a pyrolysis chamber, a deposition chamber, and a trapping chamber [9,10]. The evaporation chamber sublimates dimers (di-*para*-xylylene) at 150–200 °C, and the resulting vapor passes through the pre-heated pyrolysis chamber (600–700 °C), where the dimers are cleaved into the monomeric gaseous form. The monomeric gas is then flowed into a deposition chamber containing substrates, where it polymerizes directly on the substrate surface at room temperature. To protect the pump against parylene contamination by residual monomers, the unpolymerized monomer gases are drained to a vacuum pump following condensation by liquid nitrogen in the trapping chamber [11,12].

During preparation of transparent parylene films, the parylene deposition speed is normally controlled by the working pressure of the deposition chamber and the temperature of the evaporation chamber, to give a deposition speed of approximately 30 nm/min [13,14]. However, in the context of conformal coatings and flexible electronics, parylene films generally require a thickness of microns, thus consuming several hours of tact time. Although such a slow deposition speed results in a long tact time, a number of studies have been reported with regards to increasing the deposition rate, and characterizing the resulting fast-deposited parylene films [15,16]. Higher evaporation temperatures tend to enhance the deposition speed, causing insufficient pyrolysis, and as such, the polymerization process aggravates the transmittance of deposited parylene films. Modification of the pyrolysis chamber and

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optimization of the deposition process is therefore a priority to increase the deposition speed.

We herein report the preparation of parylene C films with a deposition speed of >500 nm/min. Analysis of the optical and thermal characteristics of these films will be compared to those of slow-deposited parylene films. UV–Vis spectrophotometry and Fourier transform infrared (FT-IR) spectroscopy will be carried out to confirm the molecular structures of the fast-deposited transparent parylene films by comparison with the slow-deposited films. In addition, the melting temperatures and melting enthalpies of the fast-deposited films will also be determined.

2. Experimental details

Parylene films were deposited using a parylene deposition system (GVTech, South Korea), and the deposition speed was controlled by control of the evaporation chamber and substrate temperatures, *i.e.*, 200 °C to 250 °C and 25 °C to -30 °C, respectively. Temperatures in excess of the conventional temperature of 175 °C in the evaporation chamber increased the dimer gas fraction and the deposition rate of parylene [12,17,18]. To achieve pyrolysis of the dimer gas, the cracking chamber was modified with an internal structure and heated to 700 °C. This additional rod which was located at the center of pyrolysis chamber guided the gas flow path through the hot wall of pyrolysis chamber, and ensured sufficient cleavage of the dimers. During deposition of the parylene film, the deposition speed was monitored using a quartz crystal monitor, confirming that deposition speeds of >500 nm/min could be achieved. Analysis of the optical and structural properties of the parylene films were measured using glass substrates which were cleaned with acetone and isopropyl alcohol prior to use. The optical transmittance and haze of the parylene films were analyzed by ultraviolet–visible–near infrared spectrophotometry (UV–Vis–NIR, Cary 5000, Agilent), FT-IR (Nicolet 6700, Thermo Scientific) and haze meter (NDH-5000, Nippon Denshoku), respectively. Structural and thermal characteristics of the parylene films were analyzed by X-ray diffractometry (XRD, Rigaku SmartLab[®]) and differential scanning calorimetry (DSC, DSC1, Mettler Toledo), respectively. Finally, surface roughness of parylene deposited by both the normal (slow) and fast deposition speeds was measured by atomic force microscopy (AFM, XE-100, PSIA).

The top-emitting OLEDs were fabricated on the parylene films with a supporting glass substrate. An evaporated silver anode was patterned through a shadow mask then multilayer structure of 5 nm thick molybdenum trioxide (MoO_3) as the hole injection layer, 73 nm thick 1,4-bis[*N*-(1-naphthyl)-*N'*-phenylamino]-4,4'-diamine (NPB) as the hole transport layer, 56 nm thick tris-(8-hydroxyquinoline)aluminum (Alq_3) as the emitting layer, 1 nm of ytterbium as the electron injection layer, and 20 nm thick Ag as cathode were deposited under the vacuum condition of 10^{-7} torr. The fabricated device was encapsulated prior to the current density–voltage–luminance (*J*-*V*-*L*) measurement. The *J*-*V*-*L* characteristics of devices were measured by a Keithley 2400 (Tektronix) semiconductor parameter analyzer and a PR-650 (Photo Research) spectrophotometer.

3. Results and discussion

Deposition of parylene films under regular (lower temperature) deposition conditions results in films exhibiting high visible wavelength transmittance as the parylene monomers polymerize gradually on the substrate surface. A rapid increase in the evaporation chamber temperature to obtain abnormally fast deposition, however, prevents cracking of the vaporized parylene dimers in the cracking chamber, giving opaque parylene films with rough

surfaces, as shown in the inset of Fig. 1 [19,20]. Likewise, a fast deposition speed over 500 nm/min could be reached only by dropping the substrate temperature, but this also resulted in the formation of opaque deposited parylene films [19,21]. The transmittance spectra of various parylene films prepared in this study are shown in Fig. 1, showing that the visible transmittance of parylene films with a glass substrate was 86.7% at the wavelength of 555 nm. The slightly lower transmittance of fast-deposited parylene films compared to slow-deposited parylene films is due to the difference in thickness of the films, which were 9.6 μm and 4 μm for fast- and slow-deposited parylene films, respectively. When compared to the transmittance of glass substrate ($\sim 90.7\%$), the actual transmittance of the parylene films was approximately 96%. It should be noted that fluctuation in transmittance originates not from the optical properties of the films, but from the waveguide of the thin film, which depends on the thickness of the films [22].

Haze, which is defined as the ratio of vertical transmittance and diffused transmittance, was then analyzed. With a parylene film of 9.6 μm thickness, the total transmittance of 86.7% was divided by the vertical and diffused transmittances of 80.4% and 6.3%, respectively. The haze of fast-deposited parylene films was measured as 7.2% when the haze of the substrate was 0.6%. Diffused transmittance and haze are directly related with the thickness of parylene films. We could therefore assume that the fast-deposited parylene films possessed few light scattering centers, such as parylene dimers or structural defects [19]. To better understand the structural characteristics of our parylene films, FT-IR and XRD measurements were carried out.

FT-IR uses a broad band of infrared light to measure the IR absorption of a molecule depending on its molecular structure. As the different chemical bonding groups absorb different wavelengths of infrared light, the FT-IR spectrum gives specific information relating to the bonds and functional groups present in the molecule of interest [23]. The FT-IR spectra of the parylene films deposited using both slow and fast deposition are compared in Fig. 2(a). The spectrum of the opaque parylene film deposited without the modified pyrolysis chamber unit is also shown for comparison. All spectra show the typical absorption peaks corresponding to parylene C in the infrared range at approximately 3000, 1500, 1050, and 815 cm^{-1} [17,24]. The weak signals at ~ 2900 cm^{-1} and ~ 3050 cm^{-1} correspond to C-H stretching in the CH_2 and aromatic

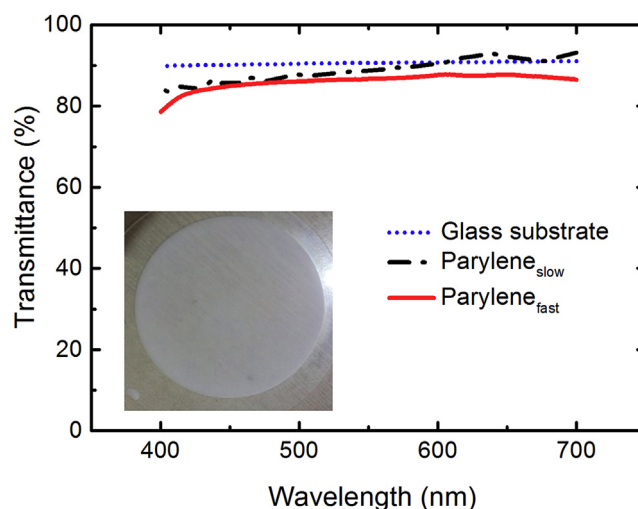


Fig. 1. Transmittance of the fast-deposited (red solid line) and slow-deposited (black dashed line) parylene films, and of the glass (blue dotted line) substrates. Inset: Photographic image of the opaque parylene film. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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