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Formation of high-purity organic thin films by gas flow deposition and the effect of impurities on device characteristics

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ABSTRACT

A gas flow deposition (GFD) system was developed to manufacture large-scale organic light-emitting diodes (OLEDs). A *N*,*N*′-di(1-naphthyl)-*N*,*N*′-diphenylbenzidine (α-NPD) thin film with a high purity of 99.97% was obtained using the GFD system. The film properties such as morphology, and electrical and optical characteristics were almost the same as those of films made by conventional vacuum thermal evaporation.

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1. Introduction

The number of products containing organic light-emitting diodes (OLEDs), mainly smart phones and tablets, has increased significantly recently, and the market is still growing rapidly. For further development of OLED technology, large-area OLEDs need to be manufactured at low cost. Manufacturing equipment aimed at minimizing production cost also have to satisfy some other important requirements including short tact time, high production yield, small footprint, film uniformity over a large area and high material utilization efficiency.

To meet these requirements, we developed a gas-flow deposition (GFD) system [1] to form consecutive multilayer organic thin films with the aim of high throughput OLED panel manufacture. A number of deposition systems using a carrier gas to control organic thin film deposition have been developed [2–5]. These deposition methods were applied to develop OLEDs, organic field effect transistors (OFETs) and organic solar cells (OSCs) toward industrial production with high throughput, large area of substrate and so on [6–8]. GFD offers a higher material utilization efficiency than these other systems and is also readily scalable because it uses linear sources.

A schematic diagram of our GFD system is presented in Fig. 1. The GFD system efficiently transports organic materials as a vapor to a substrate using an inert carrier gas, which allows the deposition rate to be regulated by precisely controlling the mass flow of the carrier gas. Advantages of using a carrier gas include low thermal stress and rapid adjustment of deposition rate compared with conventional deposition systems. Our GFD system allows different organic materials to be mixed and enables control of lower deposition rates with high precision, so it is suitable for host–guest systems. Transport using a carrier gas allows deposition sources to be placed outside the deposition chamber, which prevents cross-contamination from different sources. Carrier gas transport also allows films to form independent of substrate angle. Using this system with aligned sources enables short tact time and small footprint. However, the GFD process does suffer from several drawbacks. Organic molecules may degrade or polymerize because the pressure in the deposition sources is rather high (1–10 Pa), and the temperature of the pass-lines transporting organic vapor is same as that of the deposition sources. Thus, in this study, we investigated the degradation of a typical organic molecule, *N*,*N*′-di(1-naphthyl)-*N*,*N*′-diphenylbenzidine (α-NPD), by characterizing films of α-NPD formed by both a GFD system and conventional vacuum thermal evaporation (VTE).

2. Experimental

2.1. Conditions used to form α-NPD thin films by GFD and VTE

The conditions used to form α-NPD thin films using GFD and VTE are summarized in Table 1. In the GFD system, α-NPD (~4 g) was loaded into a source made of SUS304 stainless steel. The source temperature was kept at 290 °C and the temperature of the pass-line to a nozzle head was maintained above 290 °C to

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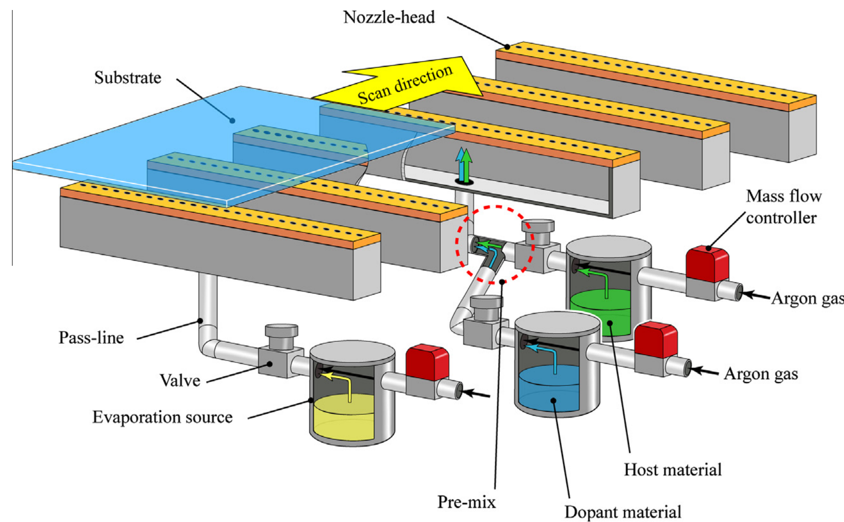

Fig. 1. Schematic diagram of the GFD system.

Table 1
Conditions used to form α-NPD thin films by GFD and conventional VTE. In the case of VTE (b), no information on heating temperature was obtained.

	GFD	VTE (a)	VTE (b)
Heating method	Heater	Heater	Resistance
Source temperature	290 °C	290 °C	–
Pass-line temperature	340 °C	–	–
Carrier gas flow	1.5 sccm	–	–
Chamber pressure	1.5×10^{-2} Pa	2.4×10^{-4} Pa	2.1×10^{-4} Pa
Source material	SUS304 source	Al_2O_3 crucible	Ta boat
Deposition rate	6.4 nm/s	1.8 nm/s	0.2 nm/s

minimize deposition inside the pass-line. Ar carrier gas was passed through the source at a rate of 1.5 sccm using a mass flow controller. The pressure of the deposition chamber was 1.5×10^{-2} Pa. α-NPD vapor was projected out from a nozzle head onto a moving glass substrate. The deposition rate was measured using a quartz crystal monitor, and maintained at a constant rate of 6.4 nm/s. In the VTE process, two typical sources consisting of an aluminum oxide (Al_2O_3) crucible and a tantalum (Ta) boat and containing approximately 1 and 0.3 g of α-NPD, respectively, were used. Film formation from the Al_2O_3 crucible was performed by heat transfer from an external heater, while that from the Ta boat was achieved by direct electric resistance heating. The temperature in the Al_2O_3 crucible was set at 290 °C, and the film was deposited at a constant rate of 1.8 nm/s [denoted VTE (a) in Table 1], whereas the Ta boat was adjusted to a target deposition rate of 0.2 nm/s [denoted VTE (b) in Table 1]. Note that the source temperature of the Ta boat was unknown. The pressures of the deposition chamber in VTE (a) and VTE (b) were 2.4×10^{-4} and 2.1×10^{-4} Pa, respectively.

For the films formed by GFD, α-NPD (~4 g) in a GFD source was heated so that the material flowed constantly from a nozzle head for at least 24 h to investigate the thermal stress on α-NPD. In this experiment, α-NPD thin films were formed on glass substrates after 2.5, 21.9 and 24.2 h. Similarly, α-NPD (30 g) was heated in the GFD system for 48 h. α-NPD thin films were formed on glass substrates after 28.5 and 36.0 h.

2.2. Evaluation of α-NPD thin films

α-NPD thin films with a thickness of about 500 nm formed on glass substrates by each process were dissolved in CH_2Cl_2. Each solution was carefully analyzed by high performance liquid chromatography (HPLC), and the purity and impurity ratio of the thin films were evaluated. HPLC was performed using acetonitrile as the mobile phase with a Shimadzu Prominence UFLC equipped with a GL Sciences Inertsil ODS-3 column (4.6 × 250 mm). Films were also characterized by atomic force microscopy (AFM). Photoluminescence (PL) spectra, refractive index and extinction coefficient were measured using Shimadzu SPM-9600, JASCO FP-6500 and Horiba UVISEL spectrometers, respectively.

A hole-only device (HOD) was fabricated to clarify the electrical properties of α-NPD thin films formed by GFD. Indium tin oxide (ITO)-coated glass substrates were washed with deionized water containing a polyvinyl alcohol sponge, ultrasonicated in deionized water for 10 min and then dried at 230 °C in a nitrogen-filled clean oven for 30 min. Substrates were treated with vacuum ultraviolet light-ozone for 10 min, and then transported under vacuum. α-NPD thin films (200 nm) were formed on substrates after 2.5 and 24.2 h of heating. An aluminum cathode was deposited by VTE through a shadow mask. After HODs were fabricated, they were

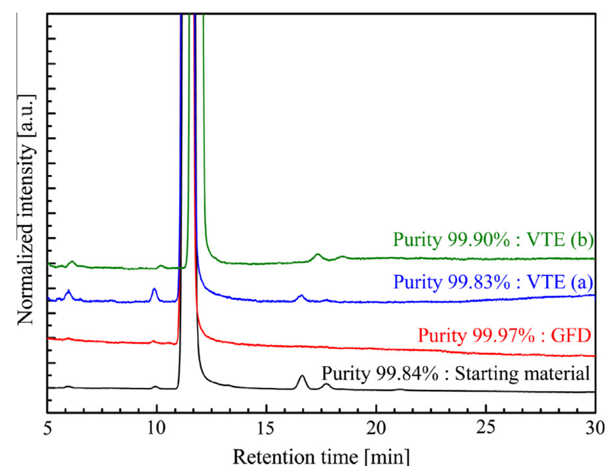

Fig. 2. HPLC results for α-NPD thin films formed by GFD, VTE (a), VTE (b) and starting material. Films of VTE (a) and VTE (b) were formed by conventional VTE using an Al_2O_3 crucible and Ta boat, respectively. The peak at about 12 min is assigned as the main peak of α-NPD. High molecular weight impurities appeared after the main peak of α-NPD, while low molecular weight ones are observed before the main peak.

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