Interface-Induced Face-on Orientation of Organic Semiconductors with a Template Layer and Its Application to Vertical-Type Organic Transistors

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Molecular orientation control with a template material, perylene 3,4,8,9-tetracarboxylic dihydride (PTCDA), was investigated for various π-conjugated crystalline molecules, namely copper phthalocyanine (CuPc), N,N’-Dimethyl-3,4,9,10-perylenedicarboximide (MePTC), pentacene and dinaphtho[2,3-b:2’,3’-f]thieno[3,2-b]thiophene (DNTT). The molecular orientation of CuPc and MePTC clearly changed from edge-on to face-on orientation by depositing on PTCDA. The electron mobility of MePTC, which is an n-type organic semiconductor suitable for vertical carrier transport, improved approximately three times owing to face-on molecular orientation. Thin-film X-ray diffraction studies revealed that the molecular packing mode (stacking or herringbone) affects the molecular orientation control. The MePTC film oriented with PTCDA was applied to a vertical-type organic transistor, a metal base organic transistor (MBOT), resulting in higher output current and current gain.

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Molecular orientation to the substrate’s surface is a fundamental problem in organic semiconductor devices owing to the anisotropic nature of the optoelectronic properties of the organic semiconductors. Organic semiconductors with planar π-conjugated cores generally show efficient charge transport along the π-π stacking direction.1 Organic molecules aggregate according to the intermolecular interactions and tend to have an edge-on molecular orientation on common substrates (e.g., indium tin oxide (ITO), glass), where intermolecular interactions are stronger than molecule–substrate surface interactions.2,4 With an edge-on orientation, molecular planes are vertical to the substrate surface, which is the same direction as the current flow. Thus, the edge-on orientation is suitable for in-plane charge transport in organic field-effect transistors (OFETs), resulting in high carrier mobility exceeding 10 cm² V⁻¹ s⁻¹.1,5,6 In contrast, a face-on molecular orientation with molecular planes vertical to the substrate is beneficial for out-of-plane charge transport, where charges flow perpendicularly to the substrate. However, it is usually difficult to induce face-on molecular orientation on the weakly-interacting substrates. This orientation problem causes much lower vertical mobility compared to the lateral mobility observed in OFETs and requires vertical-type devices to operate with an ultrathin film, which makes large-area mass production processes difficult.7,9 Moreover, the face-on orientation enables efficient light extraction in organic light-emitting diodes10 and strong photoabsorption in organic solar cells11 owing to the transition dipoles situated on the molecular plane.

Template layering techniques have been used to control the molecular orientation from edge-on to face-on without changing the molecular structure by inserting template materials between the substrate and organic semiconductor layers.1,2,9 An example is copper iodide (CuI), which changes the molecular orientation of organic semiconductors to face-on owing to the interactions between the d-orbitals of Cu and π-conjugated planes of molecules, leading to improvements in the short-circuit current densities and power conversion efficiencies in organic solar cells.12,13 Another example is 3,4,8,9-tetracarboxylic dihydride (PTCDA) in which the molecules lie parallel to the substrate’s surface. A thin PTCDA layer on the substrate indicated a templating effect to induce the face-on orientation of phthalocyanine based on π-π interactions.16-20 The reason why PTCDA molecules show the face-on orientation has been explained by strong interactions between the oxygen atoms of the carbonyl groups and the substrate and in-plane intermolecular hydrogen bonds between the hydrogen and oxygen atoms of PTCDA.21,22 However, PTCDA itself does not have good electrical properties, and the templating effect for other materials has not been studied.

In this study, molecular orientations of various organic semiconductors on PTCDA were systematically investigated in terms of the molecular structures and packings. Common crystalline molecules, pentacene, dinaphtho[2,3-b:2’,3’-f]thieno[3,2-b]thiophene (DNTT), copper phthalocyanine (CuPc) and N,N’-Dimethyl-3,4,9,10-perylenedicarboximide (MePTC), were selected as target materials for molecular orientation control. Pentacene and DNTT have linear molecular structures and herringbone molecular packings and exhibit high charge mobilities in OFETs.24,25 CuPc and MePTC have two-dimensionally extended molecular structures and slipped-stack molecular packings. To understand the impact of the PTCDA template layer on vertical mobility, the mobility of the oriented molecules was measured by the space charge limited current (SCLC) technique. Furthermore, the MePTC film oriented with PTCDA was applied to a vertical-type organic transistor, metal base organic transistor (MBOT). In the vertical-type transistors, the high carrier mobility perpendicular to the substrate was essential to achieve high current density and low voltage operation as this channel corresponds to the film thickness.8-20 The molecular orientation by the template layer is expected to improve any type of vertical organic semiconductor device.

**Experimental**

CuPc, pentacene, and DNTT purified by sublimation were purchased from Lumtec. PTCDA and MePTC were purchased from Sigma-Aldrich. CuPc, pentacene, and MePTC were purified by sublimation before the thin film fabrication. Glass and ITO substrates, 20 × 25 mm squares, were cleaned with acetone-soaked wipes, and then sonicated in acetone and isopropanol. The washed substrates were then treated with a UV-O₃ cleaner (Filgen, UV253V8). All organic molecules were deposited by thermal vacuum evaporation under a pressure of ∼10⁻⁴ Pa. The deposition rate was 1 Å/s.

The electron mobilities of MePTC films and hole mobilities of CuPc with and without PTCDA were measured by the space charge limited current (SCLC) technique. The electron-only device structure
for the mobility measurement was glass/ITO/Al (100 nm)/PTCDA (0, 2.0 nm)/MePTC (100 nm)/Al (100 nm). The hole-only device structure for the mobility measurement was glass/ITO/MoO3 (2 nm)/PTCDA (0, 0.5, 2.0 nm)/CuPc (100 nm)/MoO3 (2 nm)/Au (40 nm). The n-type metal-base organic transistor (MBOT) was fabricated using MePTC as a collector layer and CuPc as an emitter layer. PTCDA was inserted between the collector ITO electrode and the MePTC layer. Al, C60, and Ag were deposited as the base electrode, emitter layer, and emitter electrode, respectively. The device structure was glass/ITO/PTCDA (0, 2.0 nm)/MePTC (100 nm)/Al (15 nm)/C60 (30 nm)/Ag (30 nm). After deposition of the Al base electrode, the substrate was annealed at 150°C for 1 h in air. The active area of the electron-only device and the MBOT was 4 mm².

Current–voltage characteristics of the devices were measured with a semiconductor parameter analyzer (Agilent 4155C) in a nitrogen-filled glove box. The film surface morphology was observed by an atomic force microscope (AFM, Bruker, Dimension Icon) in tapping mode. The thin-film crystal structure was investigated by an X-ray diffraction meter (Rigaku, SmartLab) with out-of-plane configuration mode. The thin-film crystal structure was investigated by an X-ray atomic force microscope (AFM, Bruker, Dimension Icon) in tapping mode.

Results and Discussion

Four types of crystalline organic semiconductors with different molecular structure packings (MePTC, CuPc, pentacene, and DNTT) were deposited on the PTCDA film with 2 nm thickness as the template layer (Figures 1a and 1b). PTCDA completely covered the substrate surface above 0.5 nm. Thin film X-ray diffraction (XRD) analysis was performed in the out-of-plane configuration to evaluate the molecular orientation of the substrate with and without the PTCDA layer (Figure 2). The diffraction peaks of the out-of-plane XRD measurement corresponded to the crystallographic planes lying parallel to the substrate. CuPc is a p-type organic semiconductor having crystal structures in a one-dimensional column. The deposited films tend to prefer an edge-on orientation and are specifically suitable for OFET devices. The CuPc film on the bare substrate showed a clear peak at 13.7° (d = 6.48 Å) assigned to the (01-2) plane, indicating edge-on orientation. In contrast, on the PTCDA substrate, the (100) peak almost disappeared, and the weak peaks at 26.6° (d = 3.35 Å) and 27.7° (d = 3.22 Å) assigned to the (01-2) and (11-2) planes emerged. Both peaks represent a face-on orientation of CuPc molecules and correspond to the reported results. MePTC is an n-type organic semiconductor that shows a one-dimensional column structure. The MePTC film on the bare substrate showed a distinct peak at 12.2° (d = 7.24 Å) and a weak peak at 27.7° (d = 3.22 Å) assigned to the (001) and (102) planes. These results indicated that the MePTC molecules in the film had originally aligned partially face-on. In contrast to the CuPc film, the (001) peak completely disappeared on the PTCDA substrate, and a clear (102) peak emerged, indicating a perfect face-on orientation. The distinct XRD pattern of the MePTC film on PTCDA suggested that crystals of MePTC molecules grow more easily in the face-on state compared to CuPc molecules owing to the π-π interactions between the perylene cores of PTCDA and MePTC. Although perylene derivatives are well-known n-type organic semiconductors, MePTC is not suitable for OFET material, and structural information of MePTC films has not been widely investigated. It has been reported MePTC has a strong tendency to align face-on and has been used in vertical transistors.

High aspect ratio materials, such as pentacene and DNTT, are the most standard high performance OFET materials with a herringbone structure. The molecules easily align with the edge-on orientation, resulting in suitable carrier transport in the OFETs. Films of these materials showed peaks assigned to the (001) plane on the substrates with and without PTCDA, indicating a perfect edge-on orientation. Although pentacene and DNTT did not align face-on with PTCDA, the peak intensity of the (001) planes decreased on the PTCDA substrate. These results suggest that PTCDA suppresses the growth of the crystalline films of pentacene and DNTT with an edge-on orientation via π-π interactions.

The difference in the molecular orientation of the molecules on PTCDA was attributed to molecular packing structures in the crystals. MePTC and CuPc have slipped-stack molecular packing structures and are expected to easily maintain the π-π interactions in the vertical direction to the substrate, leading to a face-on orientation. MePTC is more easily aligned face-on than CuPc, which is preferable to vertical-type organic devices. Pentacene and DNTT have herringbone packing structures, where the molecular edges face the π-conjugated planes. Owing to this structure, maintaining the π-π interactions with PTCDA

Figure 1. (a) Structures of organic semiconductor molecules used in this work. (b) The sample structure for the thin film XRD studies. (c) The electron-only and hole-only device structure for the SCLC mobility measurements. (d) The MBOT device structure.
Figure 2. Thin film XRD patterns of (a) CuPc, (b) MePTC, (c) pentacene, and (d) DNTT with and without PTCDA. Film thicknesses of each material and PTCDA were 100 nm and 2 nm. Insert figures show molecular alignment to the crystalline planes.

is difficult, resulting in an edge-on orientation that is thermodynamically preferable.

To investigate the details of the remarkable effects of orientation control with PTCDA on MePTC, UV-vis spectra and surface morphologies were analyzed, in addition to the detailed XRD measurements. Figure 3 shows the MePTC film-thickness dependence on XRD patterns of PTCDA. The MePTC films with various film thicknesses on PTCDA showed an XRD peak of the (102) plane showing the face-on orientation (Figure 3a). It should be noted that the peak intensity of the (102) plane increased with increasing film thicknesses. This result indicated that the effect of the PTCDA template layer was maintained for films with thicknesses up to 300 nm. The UV-vis spectra (Figure 3b) indicated no changes in shape but did show a change in absorption intensity when inserting the PTCDA layer, even though the film thickness remained the same. The absorbance of the UV-vis spectra of the MePTC film increased 18% at a wavelength of 484 nm by inserting PTCDA. The incremental increase in absorbance is attributed to the orientation of the transition dipoles parallel to the substrate surface in the oriented MePTC film as the direction of the dipole moment became parallel to the electric vector of incident light. The incremental ratio changes in absorbance showed the same value (18%) for each film thickness, which indicated that the face-on orientation effect on PTCDA did not change for the different film thicknesses. This result was consistent with the XRD results.

The effects of the PTCDA layer on surface morphology were observed by AFM. It has been reported that surface morphologies of PTCDA films show small round grains with diameters of several dozens of nm.16,29 The surface morphologies of MePTC films showed changes from fiber domains with a length of 120 nm to sphere-like shapes with 62-nm diameters via the PTCDA template layer (Figures 3c and 3d). The surface of the MePTC film on PTCDA was smoother than that of the film on glass. The root mean square (RMS) value decreased from 3.27 to 2.87 nm in accordance with the morphological changes. These morphological changes were attributed to changes in the molecular orientation and diffusion of the molecules on the substrate surface. On the bare glass substrate, the π-π stacking direction of the MePTC molecules was parallel to the substrate surface with the edge-on orientation. The molecules easily diffused on the weakly interacting surface and aggregated via intermolecular interactions; then, the thin film growth preferentially occurred in the in-plane direction. In contrast, on the PTCDA film, the molecules were aligned with a face-on orientation and barely diffused on the surface, owing to the π-π interactions between the PTCDA and MePTC molecules. This means that the MePTC film on PTCDA showed out-of-plane thin film growth and high nucleation rates, resulting in the sphere-like morphology. Similar morphological changes were observed with phthalocyanine films on PTCDA.18,19

The electron mobilities of the MePTC films and hole mobilities of the CuPc films with and without the PTCDA template layer were measured by the space charge limited current (SCLC) technique. Current (I) – voltage (V) characteristics were measured on the electron-only and hole-only device structures as shown in Figure 1c. The film thicknesses of MePTC and CuPc were 100 nm. The electron and hole mobilities were estimated from the Mott–Gurney law:

\[ J = \frac{9e\varepsilon_0\varepsilon\mu V^2}{8d^3}, \]

where \( J \), \( \varepsilon_0 \), \( \varepsilon \), \( \mu \), and \( d \) represent the current density, vacuum permittivity, dielectric constant (assumed as 3.0), charge carrier mobility, and organic film thickness, respectively. The observed current of the MePTC device increased over the entire voltage range by inserting
the PTCDA template layer (Figure 4a). The $I-V$ log plots exhibited a slope of 2, indicating that SCLC was observed for the high-voltage region and electron mobility can be estimated from Equation 1. The electron mobility improved approximately three times from $1.3 \times 10^{-4}$ to $4.3 \times 10^{-4}$ cm$^2$ V$^{-1}$ s$^{-1}$. This mobility increment is attributed to the face-on molecular orientation induced by the PTCDA template layer, where the overlaps of the molecular orbitals were improved in the direction of the $\pi-\pi$ stacking. Thus, charge transport was facilitated in the vertical direction owing to the face-on orientation, resulting in an incremental increase of the vertical mobility. In contrast, the current of the CuPc devices decreased when the PTCDA template layer (Figure 4b) was inserted, and hole mobility also decreased from...
2.8 × 10−4 to 1.3 × 10−4 cm² V⁻¹ s⁻¹ for the 0.5 nm-thick PTCDA and from 2.8 × 10−4 to 5.2 × 10⁻⁵ cm² V⁻¹ s⁻¹ for the 2.0 nm-thick PTCDA, respectively, even though the molecular orientation of the CuPc changed from the edge-on to the face-on orientation. Because current and hole mobility decreased with respect to an increase in the film thickness of PTCDA, these results suggested that the PTCDA template layer can be considered as a hole blocking layer. Furthermore, as PTCDA is known to be an n-type semiconductor with a large HOMO energy barrier with CuPc, it hampers the hole transport between the CuPc and PTCDA layers (Figure 4c).28 MePTC exhibits a face-on orientation on PTCDA, which is ideal for charge transport in the vertical-type organic transistors. To improve the MBOT device, the MePTC film oriented with the PTCDA template layer was applied to the collector layer of the device (Figure 1d). The MBOT has a simple layered device structure consisting of three metal electrodes and two organic layers. The vertical stacked device structure enables high current modulation and low-voltage operation on the order of several volts, due to a channel length less than 1 μm, which corresponds to the film thicknesses of the organic layers.26,30 The electrons were injected from the emitter electrode (Ag) after applying a base voltage (V_B), which is then transmitted across the thin base electrode (Al) and collected on the collector electrode (ITO).30 The current amplification factor (h_FE) is defined as the ratio of change in the collector current (I_C) with respect to the base current (I_B) induced by the applied input voltage (V_B).26 The detailed mechanisms are explained elsewhere.26,31 The annealing process after deposition of the base electrode is necessary for the MBOT device to improve the performance.26,30 However, this process did not affect the molecular packing of MePTC because the XRD patterns of MePTC films did not change with the annealing process (Figure 5a).

The base voltage was swept from 0 to 3 V under a constant collector voltage of 5 V. The devices with and without PTCDA showed current modulation as the base voltage increased. Output (I_C – V_B) and input curves (I_B – V_B) of the MBOTs are shown in Figures 5b and 5c. The current increase of the collector current (I_C) in the off region was improved by inserting the PTCDA template layer (Table I). In addition, the PTCDA template layer suppressed the I_B. Consequently, h_FE was improved from 1241 to 3501 with increasing I_C. In the MBOT, the current flow of transmitted carriers from the base electrode was limited by the carrier transport process through the collector layer. Therefore, increased I_C is attributed to the enhancement of the vertical charge mobility of the MePTC collector layer. Additionally, the efficient charge transport in the collector layer can improve the charge carrier transmission through the base electrode, resulting in improved h_FE. Thus, the face-on orientation control with PTCDA enables efficient charge transport in the collector layer and leads to the enhancement of output current and current amplification. The off current (I_C OFF) increased from 0.31 to 3.22 mA cm⁻² by introducing the PTCDA template layer, and the on/off ratio decreased from 2179 to 263 in accordance with the incremental increase of I_C OFF. This disadvantage is also explained by the mobility enhancement.

**Conclusions**

PTCDA was inserted as the template layer between the substrate and the organic layer to induce the face-on molecular orientation of various π-conjugated molecules with different types of molecular packing structures. The MePTC and CuPc films with slip stack packing structures on PTCDA showed XRD peaks with d-spacing corresponding to the π-π stacking distances, indicating a face-on orientation. The XRD patterns of pentacene and DNTT films with the herringbone packing structure did not show any peaks indicative of face-on orientation. The vertical electron mobility of MePTC improved approximately three times owing to the face-on molecular orientation induced by the PTCDA template layer. The MePTC film oriented with the template layer was applied to the collector layer of the MBOT. The output current (I_C ON) and the current amplification factor (h_FE) were improved by inserting the PTCDA template layer, indicating that improvement of the vertical carrier mobility via molecular orientation control was an effective way to improve MBOT performance.

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**Table I. Performance parameters of the MBOT devices with and without PTCDA.**

| Device       | I_C ON (mA cm⁻²) | I_C OFF (mA cm⁻²) | h_FE | On/Off ratio |
|--------------|-----------------|------------------|------|-------------|
| MePTC        | 692             | 0.31             | 1241 | 2179        |
| PTCDA/MePTC  | 847             | 3.22             | 3501 | 263         |

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