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# High Ambipolar Mobility in a Highly Ordered Smectic Phase of a Dialkylphenylterthiophene Derivative That Can Be Applied to Solution-Processed Organic Field-Effect Transistors\*\*

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Since the 1990s, we have witnessed remarkable progress in organic semiconductor technology.<sup>[1]</sup> In particular, reasonably high carrier mobilities, exceeding those of amorphous silicon, were observed in thin-film transistors fabricated from a single crystal of rubrene.<sup>[2]</sup> In general, it is difficult to fabricate single crystals of aromatic compounds; therefore, zone-melt and Bridgeman crystal-growth<sup>[3]</sup> or vacuum crystal-growth techniques<sup>[4]</sup> are indispensable. Polycrystalline thin films are relatively easy to fabricate and suitable for practical devices. High carrier mobilities—of the order of  $1 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ —have been observed in field-effect transistor (FET) devices based on polycrystalline pentacene thin films.<sup>[5]</sup> However, defects and domain boundaries affect the carrier transport in aromatic polycrystalline thin films; therefore, the crystal growth under the vacuum process is rigorously controlled.<sup>[6]</sup>

Device fabrication with a more practical solution process has been investigated. As well as conjugated polymers,<sup>[7]</sup> precursor methods in which thin films fabricated using soluble precursors are transformed to polycrystalline thin films by thermal treatment,<sup>[8]</sup> and solution-processable pentacene and anthradithiophene derivatives, which form polycrystalline thin films using a spin-coat method, have been investigated.<sup>[9]</sup> The field-effect mobilities in these studies are of the order of  $10^{-2} \text{ cm}^2$ , and the carrier mobility is increased up to  $0.1 \approx 1 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  by thermal treatment.<sup>[8,9]</sup> The optimum mobility is lower than those of the FET devices fabricated using vacuum deposition; the device characteristics strongly depend upon the film morphology, because the organic semiconductor thin films fabricated by the solution process have many defects and exhibit low carrier mobility.

Liquid-crystalline semiconductors are also possible materials for solution-processable semiconductors. The bulk carrier-transport properties have been investigated in discotic columnar,<sup>[10,11]</sup> smectic,<sup>[12–14]</sup> nematic,<sup>[15,16]</sup> and cholesteric<sup>[17]</sup> phases by using the time-of-flight (TOF) technique and pulse-radiolysis time-resolved microwave conductivity. Recently, high hole drift mobilities exceeding  $0.1 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  have been reported in the highly ordered smectic phases of alkenylquaterthiophene and dithienylbenzene derivatives,<sup>[18,19]</sup> furthermore a high microscopic band mobility exceeding  $1 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  has also been observed in the columnar phase of hexabenzocoronene derivatives.<sup>[20]</sup>

Liquid-crystal materials are generally soluble in organic solvents because of their alkyl chains; therefore, solution-processable liquid-crystalline semiconductors have recently been synthesized and applied to light-emitting diode (LED)<sup>[21]</sup> and FET devices,<sup>[22,23]</sup> although, FET devices can also be fabricated by vacuum deposition of liquid-crystalline semiconductors.<sup>[24]</sup> In discotic systems, the zone-cast technique has been utilized for FET devices that exhibited p-type operation with a carrier mobility of the order of  $10^{-2} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ .<sup>[23]</sup> The FET devices based on crystalline thin films of liquid-crystalline oligothiophene derivatives fabricated using the spin-coat method exhibited p-type operation with an FET mobility of the order of  $10^{-2} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ .<sup>[22]</sup> In the study of FETs, the macroscopic molecular alignment in the smectic phase was retained in the crystalline phase and FET operations were carried out in the crystalline phase. These carrier mobilities were lower by one or two orders of magnitude than those of FET devices, based on polycrystalline thin films fabricated by vacuum deposition. These liquid-crystalline semiconductors crystallize at room temperature; therefore, certain defects and carrier traps were formed in the crystallization process, resulting in the shrinkage of the thin films.

One could expect that liquid-crystalline semiconductors exhibiting a highly ordered smectic phase at room temperature could develop liquid-crystalline thin films with a low defect density because of the thermal movement of liquid-crystal molecules. In this paper, we report solution-processable smectic semiconductors with fast ambipolar carrier transport. The smectic semiconductors exhibit a highly ordered smectic phase and a high hole and electron mobility in the bulk of the smectic structure at room temperature. Therefore, the homogeneous smectic thin films of liquid-crystalline semiconductors can be fabricated by the spin-coat method at room tem-

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perature. The smectic thin films were applied to FETs, which exhibited p-channel operation in the smectic phase in ambient conditions with a mobility of  $2 \times 10^{-2} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  and an on/off ratio of  $10^6$ .

It has been difficult to design liquid-crystal semiconductors that exhibit a highly ordered structure at near room temperature because the inhibition of crystallization is necessary to expand the mesomorphic temperature range; however, this also decreases the intermolecular transfer rate, resulting in low carrier mobility. In fact, terthiophene derivatives having a bulky substituent maintained the smectic phase below room temperature; however, its optimum hole mobility was  $1 \times 10^{-3} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  at best.<sup>[25]</sup> On the other hand, dialkylterthiophene and dialkylquaterthiophene derivatives without bulky substituents crystallize at around room temperature.<sup>[14,26]</sup> We have already reported that the asymmetrically substituted 5-hexyl-5''-hexynyl-2,2':5',2'-terthiophene and 5-propyl-5''-hexynyl-2,2':5',2'':5'',2'''-quaterthiophenes without bulky substituents exhibited the highly ordered smectic phases over a wide temperature range including room temperature; they also exhibited a high hole mobility exceeding  $0.1 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , determined by using the TOF technique.<sup>[18]</sup> In the highly ordered smectic phases of these liquid crystals, the molecules are closely packed; however, they do not crystallize because of their asymmetric structure. This asymmetrical substitution is effective not only in the case of alkyl-alkynyl substitution but also in other types. In this study, we synthesized an alkyl and alkylphenyl substituted terthiophene as shown in Figure 1.

2-Propyl-2,2':5',2'-terthiophene (**1**) was synthesized according to the literature.<sup>[27]</sup> **1** was brominated by *N*-bromosuccinimide. 5-propyl-5''-(4-pentylphenyl)-2,2':5',2'-terthiophene (**3**) derivatives were synthesized by using a Suzuki coupling reaction catalyzed by Pd(PPh<sub>3</sub>)<sub>4</sub> between 2-propyl-5''-bromoterthiophene (**2**) and 4-pentylphenylboric acid 2,2-dimethylpropane-diyl esters.

The phase-transition behavior and liquid-crystal phase structures of **3** were characterized by using differential scanning calorimetry (DSC), a polarized light microscope, and X-ray diffraction (see Supporting Information). The derivatives of **3** exhibited the highly ordered smectic phase at around room temperature and did not crystallize even when they were cooled to  $-50^\circ\text{C}$ . In the highly ordered smectic

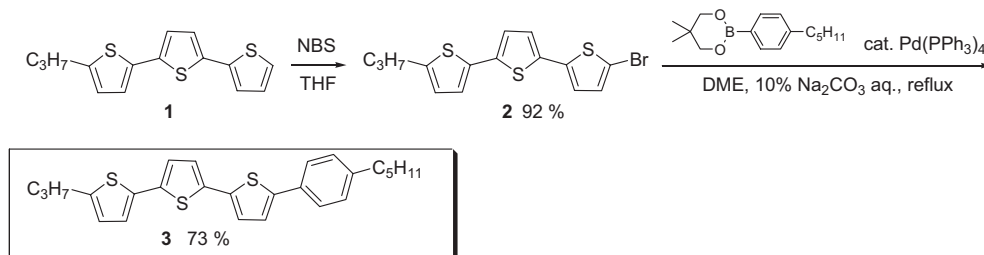
phase, liquid-crystal molecules tilted towards the normal of the smectic layers at an angle of  $57^\circ$  and the determined lattice constants within the smectic layers were shorter than those of the alkylalkynylterthiophene and quaterthiophene derivatives, which exhibited excellent hole transport properties at room temperature.<sup>[18]</sup> This result indicates that this smectic phase has a closer packing structure that is favorable for fast carrier transport. The observed domain size exceeded  $100 \mu\text{m}$ , which is much larger than those of the conventional molecular crystals.

In this study, the bulk carrier-transport characteristics were determined by using the conventional TOF technique (see Supporting Information).<sup>[28]</sup> In this study, a neat film of the compound exhibits a strong absorption band at around 400 and 250 nm in the smectic phase. The depth of the penetration of the excitation light (the third harmonic generation (THG) of the Nd:YAG (yttrium aluminum garnet) laser; wavelength  $\lambda = 356 \text{ nm}$ ) was estimated to be less than  $0.5 \mu\text{m}$ , that is, it was much smaller than the sample thickness ( $d = 25$  or  $9 \mu\text{m}$ ). The carrier mobility ( $\mu$ ) was calculated from the transit time  $t_T$ , the sample thickness  $d$ , and the applied voltage  $V$  based on Equation 1

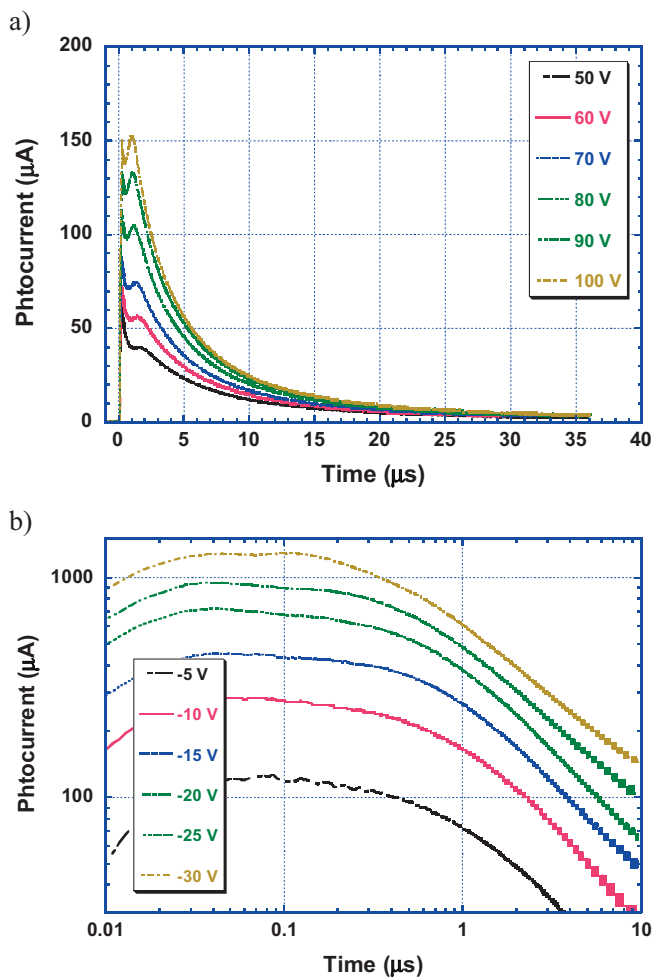
$$\mu = \frac{d^2}{Vt_T} \quad (1)$$

The highly ordered smectic phase of the compound had a relatively rigid structure with a high viscosity; furthermore, no change in the microscopic texture was observed when an electric field of the order of  $10^5 \text{ V cm}^{-1}$  was applied to the sample. The liquid-crystal molecules in the sample were aligned parallel to the electrode surface, therefore, carrier transport within a smectic layer perpendicular to the electrode surface should be observed in the TOF measurement.

Figure 2a shows transient photocurrent curves for a positive carrier at  $30^\circ\text{C}$ . For the positive carrier, clear nondispersive transient photocurrent curves were obtained above  $100^\circ\text{C}$ . Around room temperature, the initial decay before the plateau region became significant, possibly because shallow traps were formed on the defects near the illuminated electrode; however, transit times could be distinctly determined. The determined hole mobility was  $7 \times 10^{-2} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ ; this was almost independent of the electric field and temperature above



**Figure 1.** The synthetic fabrication route of 5-propyl-5''-(4-pentylphenyl)-2,2':5',2'-terthiophene. NBS: *N*-bromosuccinimide; DME: 1,2-dimethoxyethane; THF: tetrahydrofuran.



**Figure 2.** Transient photocurrent curves for a) positive carrier and b) negative carrier in the highly ordered smectic phase at 30 °C. The sample thickness was 25  $\mu\text{m}$  for the positive carrier and 9  $\mu\text{m}$  for the negative carrier, and the wavelength of the excitation pulse was 356 nm.

room temperature. This value is slightly smaller than that in the highly ordered smectic phase of the alkyalkynylquaterthiophene derivative; however, it is comparable to those of aromatic molecular crystals. Field- and temperature-independent mobilities above room temperature are typical characteristics in the smectic phases<sup>[12–14,18,19]</sup> and the discotic columnar phases of liquid-crystalline semiconductors.<sup>[10,11]</sup> Crystal-like molecular ordering and a close molecular-packing structure should reduce energetic and positional disorder as well as the hopping barrier, which results in the field- and temperature-dependence of the carrier mobility, in contrast to amorphous semiconductors. A high hole mobility is attributed to a close molecular packing in the highly ordered smectic phase and a large extension of the  $\pi$ -molecular orbital.<sup>[18]</sup>

Figure 2b shows the transient photocurrent curves for a negative carrier at 30 °C. For electrons, transient photocurrent curves with a kink point, which corresponds to the transit time, were observed, although they were slightly dispersive and the current tails were slightly long. This is because of the

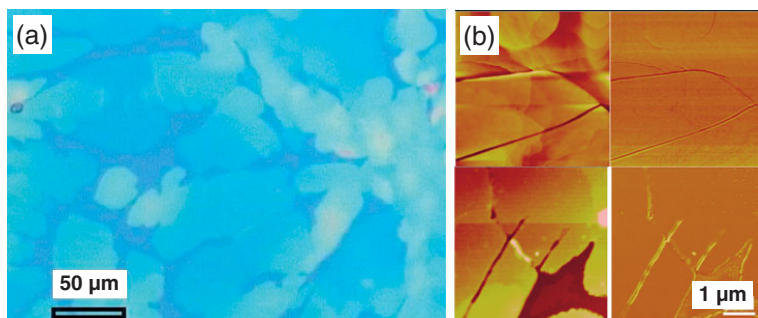
shallow traps formed by oxygen and impurities. From the kink points, electron mobility was estimated to be  $0.2 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ . The mobility was almost independent of sample thickness; therefore, we can exclude the possibility that the transient photocurrent curves were caused by hole transport from the carrier-generation region to the illuminated electrode. This electron mobility is comparable to those of molecular crystals and the optimum value among liquid-crystalline semiconductors, including smectic and columnar systems. Above room temperature, the electron mobility was almost independent of the temperature and electric field. This behavior was the same as that of the hole and typical for the smectic phase of liquid-crystalline semiconductors.<sup>[12–14,18]</sup> Closer molecular packing and a highly ordered structure should enhance the intermolecular charge transfer, resulting in high electron mobility.

The electron mobility was higher than that of the hole in this phase of the compound. In inorganic semiconductors, in which electric conduction can be described with the band scheme, the electron mobility is higher than that of the hole because the latter has a larger effective mass than the electron. In organic semiconductors, the band scheme is not always valid but the charge-carrier-hopping or polaron-hopping mechanism is more plausible.<sup>[29]</sup> In these mechanisms, which have localized charge carriers, the rate-determining process is the intermolecular charge transfer, which is determined by the intermolecular transfer integral. In oligothiophene derivatives, the lowest unoccupied molecular orbital (LUMO) has a large intensity on S atoms, whose van der Waals radii are larger in contrast to the highest occupied molecular orbital (HOMO), which is located mainly on the carbon framework. As a result, the intermolecular  $\pi$ -orbital overlap is large for the LUMOs.<sup>[30]</sup>

Various organic semiconductors have been designed and synthesized; however, the number of those that exhibit high electron mobility is currently quite limited (for example, alkylated fullerene derivatives were applied to FET devices with high electron mobility<sup>[31]</sup>). This ambipolar carrier-transport property will enable the fabrication of complementary metal oxide semiconductor (CMOS) devices such as inverters using liquid-crystalline semiconductors. Oligothiophene derivatives have been recognized as p-type semiconductors, however, we already have ambipolar carrier transport in the smectic phases of dialkylterthiophene derivatives.<sup>[14]</sup> Recent results reveal that an organic semiconductor such as polythiophene can be ambipolar if the density of trapping sites, such as defects and impurities in the bulk or interface, are sufficiently reduced.<sup>[32]</sup> Our results also support this current view.

This liquid-crystalline semiconductor can be applied to the thin-film fabrication of the solution process because of the excellent carrier transport characteristics in the bulk. The chlorobenzene solution of this compound (0.6 wt %) was spun on a  $\text{SiO}_2/\text{Si}$  substrate at 1500 rpm for 25 s forming liquid-crystalline thin films with a thickness of 50 nm.

From the polarized-light microscope observation, shown in Figure 3a, the thin films comprised a polydomain structure, whose size was of the order of several tens of micrometers.



**Figure 3.** The optical texture and atomic force microscopy (AFM) image of the smectic thin film of 5-propyl-5'-(4-pentylphenyl)-2,2':5',2'-terthiophene. a) The optical texture under polarized light microscope. b) The tapping-mode AFM images: the top images are before annealing, and the bottom images are after thermal-annealing at 120 °C for 15 min. The left side shows the topographical image, and the right side shows the phase image.

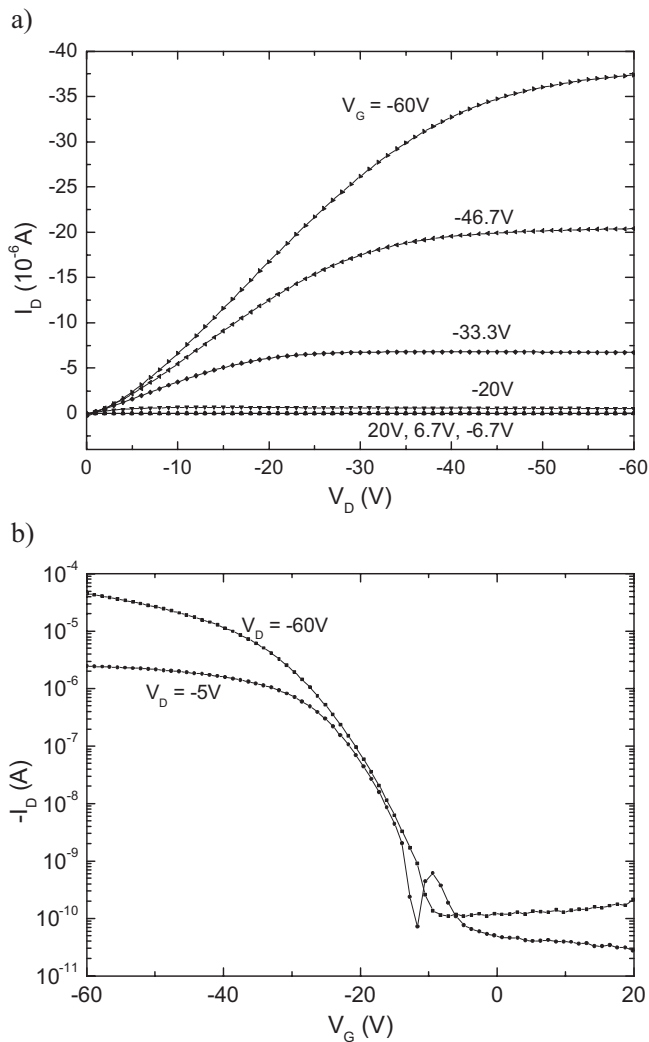
Each domain was optically anisotropic indicating that it was not amorphous but was a phase with molecular order, such as a crystal phase or mesophase. In the out-of-plane X-ray diffraction of thin films, a sharp diffraction peak at the angle  $2\theta = 3.9^\circ$  in the low-angle region was observed, indicating that the films were in the smectic phase. The layer spacing was almost the same as that of the bulk state. In the high-angle region of in-plane diffraction, only a broad halo of approximately  $21^\circ$  was observed. This X-ray diffraction indicated that this thin film had a clear layer structure but not a long-range order within layers, and that the thin film should exhibit a smectic phase. Because of the stronger diffraction intensity associated with layer spacing than those related to the order within layers, the liquid-crystal molecules aligned almost perpendicularly to the substrate surface with a small tilt angle. According to atomic force microscopy (AFM) observation, the surface of the films was not flat but showed a roughness of the order of several nanometers, as shown in the photographs at the top of Figure 3b. However, following the thermal-annealing of thin films at 120 °C for 15 min in a vacuum oven, the surface morphology was remarkably modified. The photographs at the bottom of Figure 3b show an AFM observation of the surface of the thin films after annealing. The surface was completely flat on the molecular scale. This indicated that the domain had a highly ordered structure and low defect density. The domain size was 10–50  $\mu\text{m}$ , which is comparable to the channel length (20–30  $\mu\text{m}$ ) of the normal organic field-effect transistor (OFET) structure; we can expect that the characteristics of the devices based on this thin film will be good. In comparison to conventional molecular-crystal thin films, large and flat grains were formed.<sup>[33]</sup> In the in-plane X-ray diffraction following thermal-annealing, a weak but sharp peak at  $2\theta = 19.15^\circ$  was observed, indicating a molecular order within the layers. This angle was almost the same as the strongest diffraction peak in the high-angle region of the bulk state. This result also indicated that thermal-annealing promoted molecular alignment. The diffraction peaks of the thin films were too weak to study detailed structures; however, the

structure of the thin film should be similar to the bulk sample. It should be noted that the freedom of molecular movement, such as rotation around the molecular axis and translational diffusion within smectic layers in the liquid-crystalline smectic phase, should promote the reorganization of molecular alignment in the thin films, affecting the formation of high-quality thin films and more rapid morphology change of the thin films than that of polycrystalline films. This is in addition to the high bulk carrier mobility caused by the molecular ordered structure in the smectic phase.

This result of the formation of high-quality smectic thin films motivated us to fabricate thin-film transistors. The Au source and drain electrodes were vacuum-deposited on the annealed thin films using a shadow mask. The gap between the source and drain electrodes was 20–30  $\mu\text{m}$  and the channel width was 5 mm. In the measurement under ambient conditions, a clear p-type operation of devices was observed. As shown in Figure 4a, saturation was observed in the output characteristics—the graph of source–drain current versus source–drain voltage. A superlinear behavior of the source–drain current in the case of low source–drain voltage should be obtained because of the contact resistance between the source–drain electrode and the liquid-crystal layer. The hole mobility was calculated to be  $0.02 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  and the on/off ratio was about  $10^6$  in the saturation regime from Figure 4b. This value is smaller than those of FET devices based on organic polycrystalline thin films, however, it is the best value among the devices based on solution-processed liquid-crystalline semiconductors, including smectic<sup>[22]</sup> and discotic systems.<sup>[23]</sup> Good characteristics can be obtained from a highly ordered structure of this thin film and a larger grain size than the channel length. The obtained field-effect mobility of the positive carrier was slightly lower than that determined using the TOF technique. Field-effect mobility is usually lower than that determined from the TOF technique because it is calculated based on the source–drain current affected by other factors (such as contact resistance, imperfect coverage of the active layer between the channel areas, and carrier traps at the surface of the dielectric layer) rather than the carrier-transport process. In this case, the coverage in the channel area was not perfect and the domains were not oriented. The mobility can be further increased by the optimization of the film-formation conditions and control of the domain alignment.

In this study, the n-channel operation was not observed because the measurement was carried out under ambient conditions; therefore, the device characteristics should be affected by oxygen and moisture. In addition, the Au source and drain electrodes made it difficult to inject electrons into the liquid-crystalline thin films. At this time, the fabrication of devices with source and drain electrodes with a low work function, such as Al/LiF under an inert atmosphere, is in progress.

In summary, a new liquid-crystalline semiconductor phenylterthiophene derivative was synthesized. It exhibited ambipo-



**Figure 4.** a) The output characteristics and b) transfer characteristics of the thin-film transistors based on the smectic thin film of 5-propyl-5''-(4-pentylphenyl)-2,2':5',2'-terthiophene. The device was fabricated on a Si/SiO<sub>2</sub> substrate in which the oxide-layer thickness was 300 nm. The source and drain electrodes were made of Au (thickness 60 nm). The channel length and width were 20 μm and 5 mm, respectively. They were measured under ambient conditions at room temperature.

lar carrier transport in the bulk in the highly ordered smectic phase. With the TOF technique, hole and electron mobilities were 0.07 and 0.2 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, respectively. Smectic thin films with a thickness of 50 nm were fabricated on SiO<sub>2</sub>/Si substrates with a spin-coating method. Large domains, larger than several tens of micrometers, with flat molecular scale, were formed after thermal-annealing. Thin-film transistors were fabricated based on the smectic thin films, which exhibited a p-channel operation with a mobility of 0.02 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> under an ambient atmosphere. This is the best value of mobility among transistors based on liquid-crystalline semiconductors fabricated by using the solution process.

## Experimental

**Time-of-Flight Measurement:** The setup consisted of a pulse laser (THG of a Nd:YAG laser. Laser photonics, Minilaser: wavelength, 355 nm; pulse duration, 1 ns) for excitation, the sample on a hot stage, a serial resistor, and a digital oscilloscope (Tektronics MD-600B). All the measurements were carried out under atmospheric conditions. The sample comprised conventional liquid-crystal cells consisting of two indium tin oxide (ITO) coated glass plates spaced by silica particles. The sample was fixed on a hot stage whose temperature was controlled by a PID thermocontroller with an accuracy of 0.1 °C.

**Fabrication and Characterization of Thin-Film Transistors:** A chlorobenzene solution of 5-propyl-5''-(4-pentylphenyl)-2,2':5',2'-terthiophene (0.6 wt %) was spun on SiO<sub>2</sub>/Si (the thickness of SiO<sub>2</sub> layer, 300 nm) at a rotation rate of 1500 rpm for 25 s. Prior to the spin-coating, the surface of the SiO<sub>2</sub> layer was treated with hexamethyldisilazane to make it hydrophobic. After film deposition, the sample was dried and annealed in the vacuum oven at 100 °C for 10 min. The Au source and drain electrodes (thickness, 60 nm) were deposited under a high vacuum (pressure: 10<sup>-4</sup> Pa; deposition rate: 1 Å s<sup>-1</sup>). The transistor characteristics were measured using a two-channel source meter (Keithley source meter 2612) in a light-shielded box under ambient condition with low humidity at room temperature. The surface morphology of the thin films was measured in air with a Veeco Dimension 3100 atomic force microscope.

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