

# Interfacing Solution-Grown C<sub>60</sub> and (3-Pyrrolinium)(CdCl<sub>3</sub>) Single Crystals for High-Mobility Transistor-Based Memory Devices

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Organic field effect transistors (FETs) based on organic single crystals are ideal candidates for high-performance transistor-based memory devices due to their high charge mobility; however, they have not been largely considered for memory devices due to the practical difficulty in interfacing organic single crystals with memory functional materials such as ferroelectrics. Here, we demonstrate that well-aligned ferroelectric single crystals of (3-pyrrolinium)(CdCl<sub>3</sub>) can be prepared, from solution, on top of well-aligned semiconducting C<sub>60</sub> single crystals, using an orthogonal solvent. By showing a large memory window of  $66 \pm 7$  V as well as a high electron mobility of  $1.28 \pm 0.41$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, these bilayered single crystals are potentially useful for high-performance FET memory devices with high operation speed.

Organic FETs are promising for nonvolatile memory applications, offering varied advantages including nondestructive readout, low-cost solution processability, mechanical flexibility, and direct integrated circuit architectural compatibility.<sup>[1–5]</sup> The charge carrier mobility, governing the operation speed of FETs, is a critical parameter in memory devices.<sup>[6,7]</sup> Organic single-crystalline semiconductors have exhibited promising charge transporting performance due to few defects and highly ordered structures they occupied.<sup>[8–10]</sup> Ideally, integrating single crystals of organic semiconductors into memory devices as an active layer is desired because high mobility is expected. However, this strategy is practically challenging and thus less-frequently considered<sup>[11,12]</sup> as it requires to interface organic single crystals and information storage elements such as the widely

used ferroelectric materials,<sup>[7,12–22]</sup> electrets,<sup>[23–25]</sup> and other gate materials with electron storage functions.<sup>[4,6,16,26–30]</sup> Only in two cases<sup>[11,12]</sup> were single crystals of 6,13-bis(triisopropylsilylethynyl) pentacene randomly deposited on poly(vinylidene fluoride-trifluoroethylene) [P(VDF-TrFE)] thin films to fabricate ferroelectric FET memory devices with hole mobilities below 1 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>. In order to further improve the performance of the single-crystal-based devices, a more controllable crystallization method to interface organic single crystal and the memory functional materials is needed. Recently, a facile droplet-pinned crystallization (DPC) method has been developed to prepare, from solutions, well-aligned organic single crystals.<sup>[31,32]</sup> Memory properties may be implemented in these well-organized single crystals. In principle, this DPC method is not limited to the crystallization of organic semiconductors. We envision that if the DPC method can be used to crystallize a soluble inorganic memory functional material such as molecular ferroelectrics,<sup>[33,34]</sup> it should be possible to interface organic semiconducting single crystals with inorganic ferroelectric crystals to realize high-performance memory devices using orthogonal solvents. In this work, we demonstrated this approach by growing single crystals of ferroelectric organometal halide perovskite,<sup>[35]</sup> on high-mobility C<sub>60</sub> single crystals.<sup>[36–38]</sup> FETs based on these hybrid bilayered crystals exhibited a large memory window of  $66 \pm 7$  V as well as high electron mobility of  $1.28 \pm 0.41$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>.

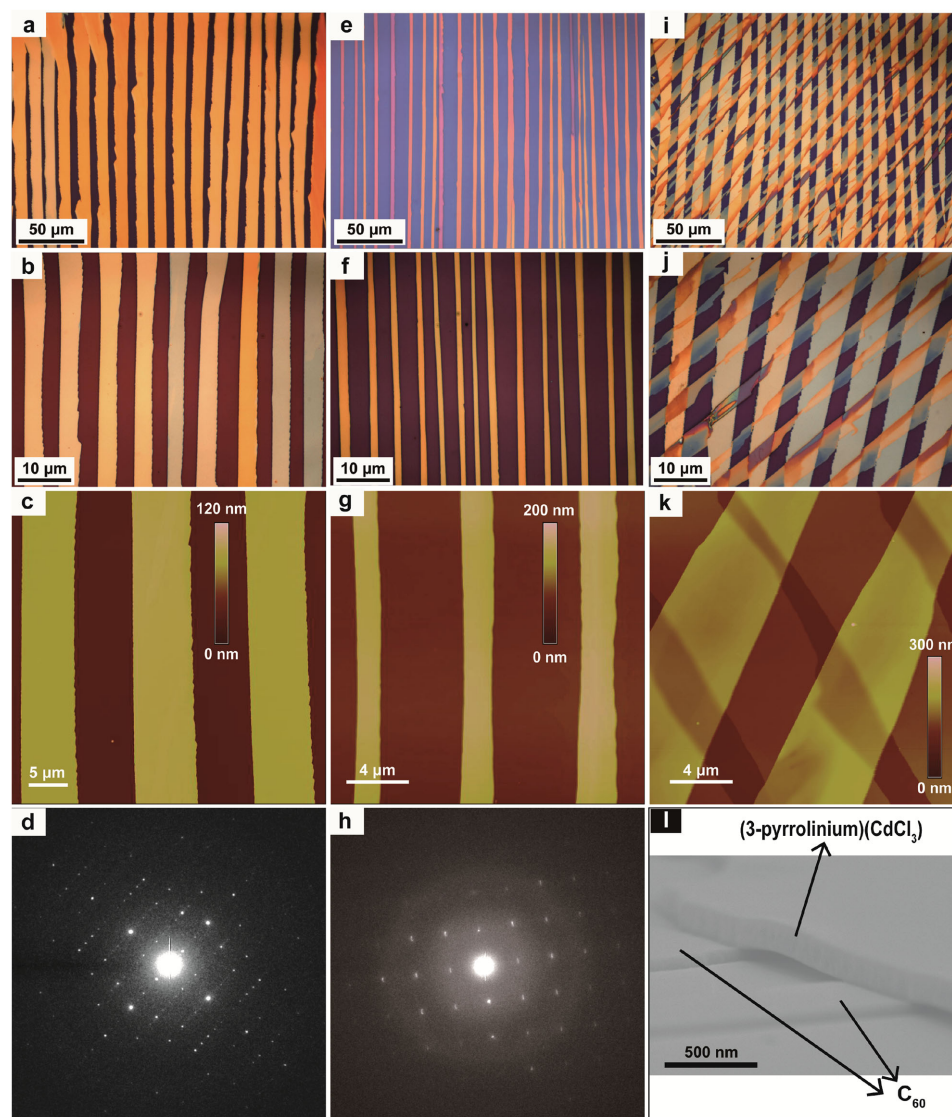
(3-Pyrrolinium)(CdCl<sub>3</sub>) was selected as the ferroelectric material and its above-room-temperature ferroelectric properties were previously reported.<sup>[35]</sup> Similar to the crystallization of varied organic semiconductors,<sup>[31,32,39–41]</sup> receding of a pinned droplet containing (3-pyrrolinium)(CdCl<sub>3</sub>) solution in ethanol resulted in well-aligned crystals (Figure 1e,f). A thickness of  $86.5 \pm 6.9$  nm and a width of  $2.2 \pm 0.5$  μm were measured from 25 crystals using atomic force microscope (AFM) (Figure 1g). X-ray diffraction showed that the crystallographic structure of these ribbons was consistent with that previously reported (Figure S1, Supporting Information).<sup>[35]</sup> Furthermore, selected area electron diffraction (SAED) indicated the single crystallinity of the (3-pyrrolinium)(CdCl<sub>3</sub>) ribbons by showing a single set of diffraction spots (Figure 1h). Consistently, these ribbons in between cross polarizers exhibited the same color and brightness, as a supporting evidence for the single crystallinity (Figure S2).

Next, (3-pyrrolinium)(CdCl<sub>3</sub>) single-crystalline ribbons were grown on top of well-aligned C<sub>60</sub> single-crystalline ribbons to prepare bilayered single crystals. Very recently, we reported

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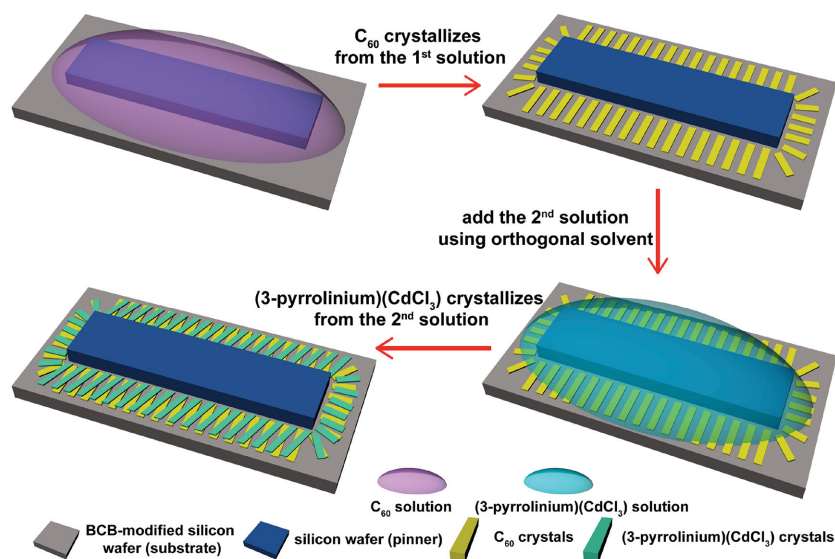


**Figure 1.** The morphologies and crystalline structures of C<sub>60</sub> crystals (a–d), (3-pyrrolinium)(CdCl<sub>3</sub>) crystals (e–h), and their bilayered heterojunctions (i–l), respectively. a,b,e,f,i,j) Optical microscopy (OM) images; c,g,k) AFM images; d,h) SAED patterns showing single sets of the diffraction spots. i) A scanning electron microscope (SEM) image (side view) of the bilayered heterojunctions.

that single-crystal bilayers of organic semiconductors formed through a sequential crystallization fashion from a mixed solution of two molecules.<sup>[42,43]</sup> However, this method does not apply to the pair of (3-pyrrolinium)(CdCl<sub>3</sub>) and C<sub>60</sub> because a common solvent and, thus, a mixed solution are not available due to the distinct molecular polarity. Instead, two solutions were used to crystallize in two steps in this case, as described in **Figure 2**. First, the C<sub>60</sub> ribbons were prepared using the previously reported<sup>[32]</sup> DPC method (Figure 1a–c) and the single crystallinity of these ribbons was reconfirmed by SAED (Figure 1d). Subsequently, (3-pyrrolinium)(CdCl<sub>3</sub>) solution in ethanol was dropped on the C<sub>60</sub> ribbons. During the slow evaporation of ethanol, the droplet receded across the arrays of C<sub>60</sub> ribbons without dissolving the C<sub>60</sub> crystals. Instead, a new layer of aligned (3-pyrrolinium)(CdCl<sub>3</sub>) formed on top of the intact C<sub>60</sub> layer. Top view (Figure 1i–k) and the side view (Figure 1l) images clearly

show the bilayered structure. Measured from 25 bilayers using AFM, the thicknesses are  $(92.8 \pm 46.8) \text{ nm}/(45.0 \pm 12.5) \text{ nm}$  and the widths are  $(4.5 \pm 1.5) \mu\text{m}/(7.1 \pm 1.6) \mu\text{m}$  for the top/bottom layers, respectively.

After obtaining the bilayered single crystals, we proceeded to fabricate FETs (**Figure 3f** inset) and examined their memory and charge transport properties in an N<sub>2</sub> glovebox. Sixty devices were examined and a large threshold voltage ( $V_T$ ) shift of  $66 \pm 7 \text{ V}$  was observed, when cycling the gate-source voltage ( $V_G$ ) between  $-100$  and  $100 \text{ V}$  at a drain voltage bias ( $V_{DS}$ ) of  $50 \text{ V}$  (Figure 3a, black dot). This large threshold shift was induced by the addition of (3-pyrrolinium)(CdCl<sub>3</sub>) ferroelectrics on the C<sub>60</sub> semiconductors. As FETs based on only C<sub>60</sub> single crystals were examined under the same  $V_G$  cycling, almost no  $V_T$  shift in transfer curves was observed (Figure 3a, red dot). This large threshold shift and the associated hysteresis can be used for



**Figure 2.** Schematic representations of the two-step crystallization process. A  $C_{60}$  solution is first dropped on a BCB-modified silicon wafer with a smaller piece of wafer to pin the droplet. As the solvent evaporates and the droplet recedes slowly,  $C_{60}$  molecules crystallize along the receding direction of the droplet. Subsequently, adding another droplet of (3-pyrrolinium)( $CdCl_3$ ) solution in an orthogonal solvent results in bilayered crystals after the (3-pyrrolinium)( $CdCl_3$ ) crystals form on top of the  $C_{60}$  crystals. Crystallization in the top layer is affected by the morphology of the bottom layer and the orientation of the crystals is determined by the receding direction of the droplet as well as the surface topology. As a result, the crystal orientations in the top and bottom layers are not identical.

organic memory devices.<sup>[16]</sup> In addition, the FET performance was studied in the saturation regime. Excellent gate modulation was observed in the typical transfer and output characteristics (Figures 3b and Figure S4, Supporting Information). An average electron mobility ( $\mu_e$ ) of  $1.28 \pm 0.41 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  (Figure 3c), on-to-off current ratios ( $I_{on}/I_{off}$ )  $> 10^5$  and  $V_T$  between 30 and 60 V were achieved. The mobility is comparable to that of FETs based on only  $C_{60}$  single crystals with a  $\mu_e$  of  $1.40 \pm 0.47 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  (Figure S2b, Supporting Information).<sup>[32]</sup> Therefore, the introduction of the ferroelectric layer induced the memory window without dramatically reducing the charge mobility.

Ferroelectrics have been widely used in FET-based memory devices where they act as the dielectrics as well.<sup>[16,17,21]</sup> Since charges are transported at the dielectric/semiconductor interface, the quality of the ferroelectrics, such as surface roughness, affects greatly the charge mobility. For example, ferroelectric polycrystalline P(VDF-TrFE) films were commonly used as the gate dielectric. The mobility of obtained devices was, however, relatively low partially because of the charge trapping on the rough surface of P(VDF-TrFE) dielectrics.<sup>[7,44]</sup> In order to reduce the effects of ferroelectrics on the charge transport, the ferroelectric crystals were stacked on the top surface of the semiconducting layer while the conducting channels were formed on the bottom in this work. As a result, the high electron mobility of the  $C_{60}$  semiconducting layer was retained after the ferroelectrics were introduced.

To further investigate the memory characteristics, write-read-erase-read (WRER) cycling and the retention time test were carried out. The WRER curve (Figure 3d), by applying gate voltages of  $\pm 100 \text{ V}$  programmed/erased pulses and drain voltage

at 50 V, shows the endurance characteristics of the memory devices based on the bilayered single crystals. The currents in between ON and OFF states (at zero gate bias) differ by more than three orders of magnitude and this high memory ratio maintains for at least 100 cycles, indicative of reversible and stable switching behaviors with a good stress endurance performance (Figure 3e). Figure 3f is the result of the retention time test, where the ON and OFF states were measured at a time interval of 50 s at zero gate bias and  $V_{DS} = 50 \text{ V}$  after applying the erased/programmed bias voltage of  $\pm 100 \text{ V}$  for 1 s. The two states maintain for long retention time of at least  $10^4 \text{ s}$ .

In summary, aligned ferroelectric single crystals of (3-pyrrolinium)( $CdCl_3$ ) were grown, from solutions, onto aligned semiconducting  $C_{60}$  single crystals using an orthogonal solvent. FETs based on the obtained ferroelectric/semiconductor bilayered single crystals exhibited a large memory window ( $V_T$  shift of  $66 \pm 7 \text{ V}$ ), a long retention time, a high cycle endurance, and, equally importantly, a high electron mobility of  $1.28 \pm 0.41 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ . Interfacing the molecular ferroelectric single crystals with organic semiconducting single crystals through the

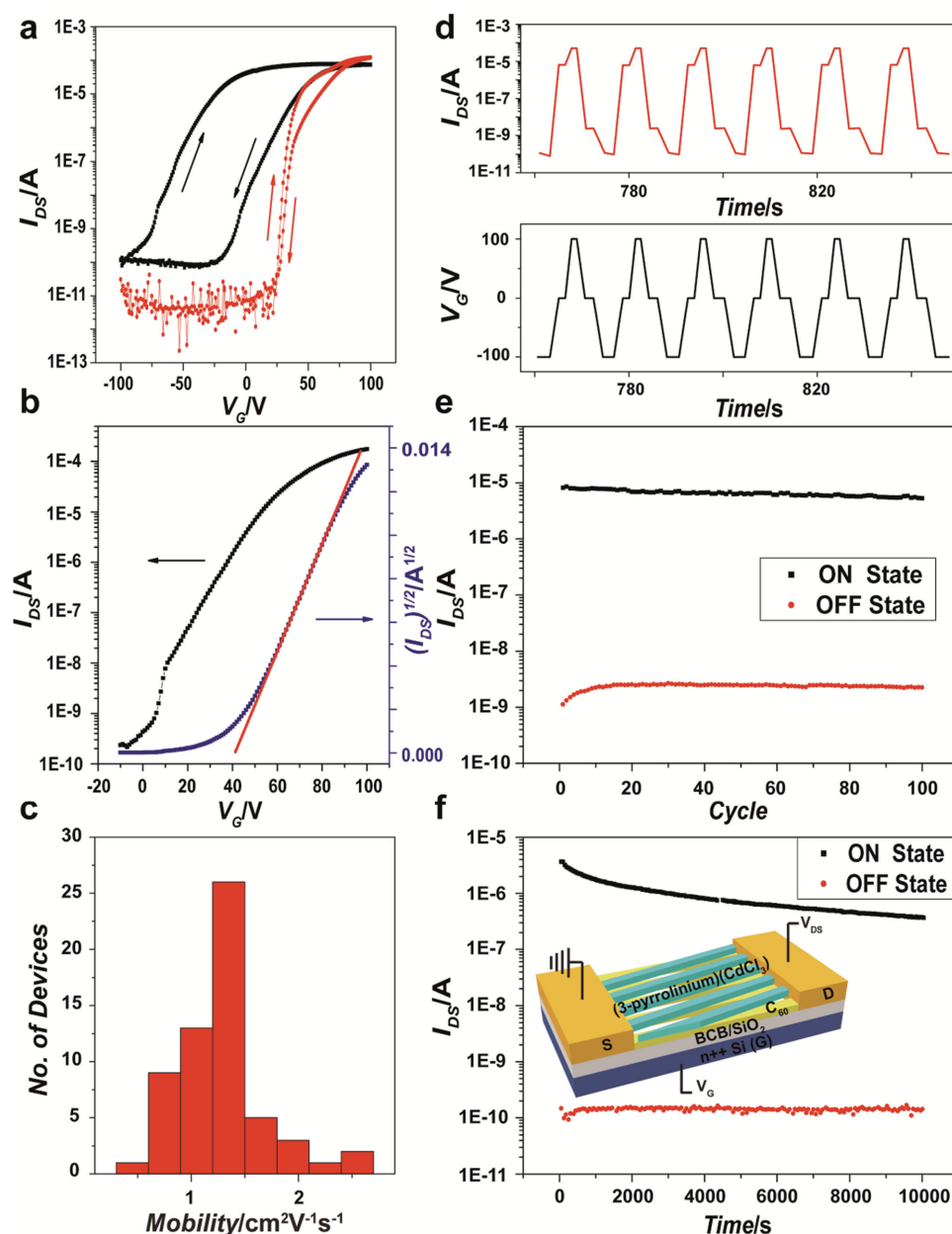
solution growth method provides a facile approach to fabricate high-performance FET-based memory devices. Expanding the material systems to construct varied multilayered highly crystalline films<sup>[45]</sup> should further help realization of multifunctional FETs<sup>[46]</sup> based on organic single crystals.

## Experimental Section

(3-Pyrrolinium)( $CdCl_3$ ) was synthesized according to the literature procedure.<sup>[35]</sup>  $C_{60}$  (Alfa Aesar) and (3-pyrrolinium)( $CdCl_3$ ) single crystals were prepared on a piece of highly doped silicon substrate ( $1 \text{ cm} \times 1 \text{ cm}$ ) modified by divinyltetramethyldisiloxane bis(benzocyclobutene) (BCB) (Dow Chemicals), which was spin coated from a mesitylene (Fluka) solution ( $V_{BCB}:V_{mesitylene} = 1:30$ ) and then thermally cross-linked after heating in a  $N_2$  glovebox. (3-Pyrrolinium)( $CdCl_3$ ) was dissolved in ethanol (Aladin) and  $C_{60}$  was dissolved in mixed solvents of *m*-xylene (Sigma-Aldrich) and carbon tetrachloride ( $CCl_4$ ) (Aladin) ( $V_{CCl_4}:V_{m-xylene} = 4:3$ ) with the concentrations of 0.8 and  $0.4 \text{ mg mL}^{-1}$ , respectively. A solution ( $20 \mu\text{L}$ ) was transferred onto the substrates with a piece of silicon wafer ( $0.4 \text{ cm} \times 0.4 \text{ cm}$ , pinner) to pin the solution droplet. The substrates were placed on a Teflon slide inside a Petri dish ( $35 \text{ mm} \times 10 \text{ mm}$ ) sealed by parafilm on a hotplate of  $30 \pm 1^\circ \text{C}$ . For bilayered crystals, the  $C_{60}$  solution ( $40 \mu\text{L}$ ) was first dropped on the substrate ( $1 \text{ cm} \times 2 \text{ cm}$ ) with a longer pinner ( $0.4 \text{ cm} \times 1.6 \text{ cm}$ ). Subsequently, (3-pyrrolinium)( $CdCl_3$ ) crystals were grown on the  $C_{60}$  crystals from a droplet ( $50 \mu\text{L}$ ).

The morphologies of the crystals were characterized by OM (Nikon LV100 POL), AFM (Veeco 3D), and SEM (Hitachi S4800 field-emission SEM System). The single-crystalline structures were examined by SAED (JEOL 1400). Bottom-gate, top-contact configuration FETs were constructed in a bottom-gate and top-contact configuration by depositing source and drain electrodes ( $80 \text{ nm Au}$  for  $C_{60}$  single





**Figure 3.** Charge transport characteristics of the (3-pyrrolinium)(CdCl<sub>3</sub>)/C<sub>60</sub> bilayered single crystals. a) Hysteresis characteristics of FETs based on C<sub>60</sub> crystals with (black dot) and without (red dot) a layer of (3-pyrrolinium)(CdCl<sub>3</sub>) crystals on top. b) Typical transfer characteristics of the devices. c) Histogram of electron mobility. d,e,f) WRER cycles, endurance characteristics, and retention time test of the memory devices, respectively. The inset in (f) is a schematic diagram of the FET configuration, where S is the source, D is the drain, and G is the gate.

crystals and 150 nm Au for bilayered single crystals), with channel length ( $L$ ) of 50  $\mu\text{m}$ , width ( $W$ ) of 1 mm, and  $W/L$  of 20. Device performance was measured in an N<sub>2</sub> glovebox using a Keithley 4200-SCS semiconductor parameter analyzer. The real  $W/L$  value needs to be measured for the mobility calculation, but this measurement is technically very difficult because of the bilayered structure. Instead, the  $W/L$  value of 20 was used for all the mobility calculation and the obtained mobility values were, therefore, slightly underestimated because the C<sub>60</sub> crystals did not completely cover the whole channel of the devices. For statistics, data of crystal widths, thicknesses, and mobility are displayed as mean  $\pm$  SD.

## Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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