Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

Supporting Information

Two side chains, three supramolecules: exploration of fluorenone derivatives towards crystal engineering

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Synthesize:

At first, the commercially available 2,7-bis(10-ethoxycarbonyldecyloxy)-9-fluorenone was used, with $BrC_{14}H_{29}$ in acetone (60 °C, 50 mL) along with K_2CO_3 added. Four hours later, we obtained the mono-substituted fluorenone derivative, with a yield of 52%. Then the mono-substituted fluorenone derivative was used, together with BrC_7H_{15} in DMF (150 °C, 50 mL) along with K_2CO_3.The reaction finished twelve hours later and proved to be quite successful with a yield of 99%. The desired products were obtained by repeated recrystallization for the sake of high degree purity. The solvent was purchased from Tokyo Chemical Industry (TCI) without further purification.

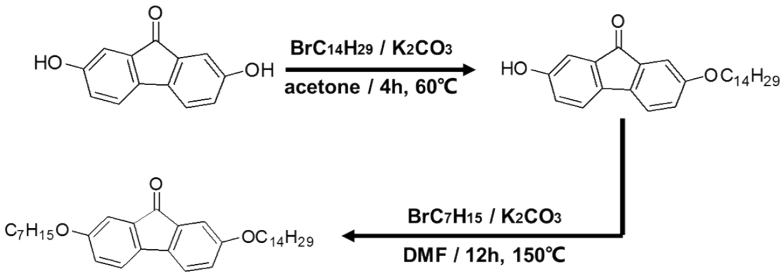

Fig. S1 Synthesis of F-$C_{14}C_7$.

Separation and purification methods

The F-C_7C_7, F-$C_{14}C_7$ and F-$C_{14}C_{14}$ were obtained after the reaction finished. Then the separation was conducted through the method of column chromatography. Next, the obtained compounds were dissolved in dichloromethane under the concentration close to saturation. Then ethyl alcohol was added into the solution drop by drop. About 5 minutes later, the compounds separated out from the solution, and then they were filtered and dried in vacuum oven.

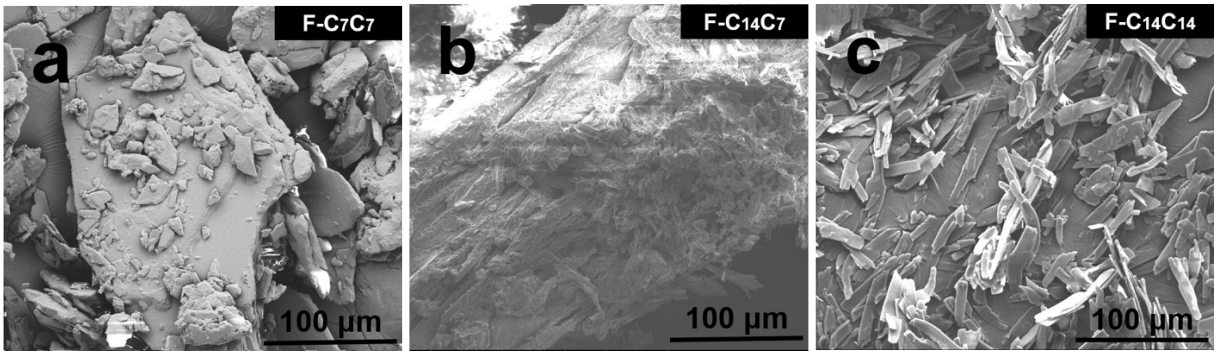

Fig. S2 Low-magnification SEM images for compounds F-C_7C_7, F-$C_{14}C_7$ and F-$C_{14}C_{14}$.

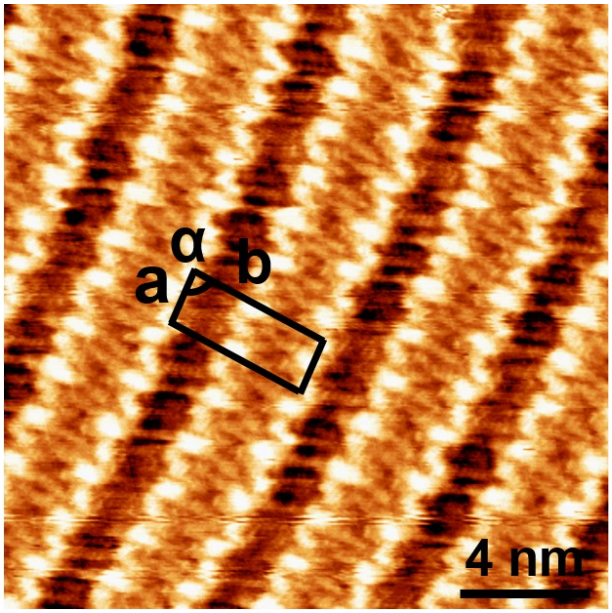

Fig. S3 High-resolution STM image of Plier-like structure for F-$C_{14}C_7$ at the *n*-tetradecane/HOPG interface (concentration: $C_0 = 1.5 \times 10^{-6}$ M). Scanning conditions: $I_t = 580$ pA, $V_{bias} = 720$ mV. The unit cell parameter are $a = 1.5 \pm 0.1$ nm, $b = 5.5 \pm 0.1$ nm, $\alpha = 88 \pm 1°$. Every unit cell consists of four molecules and the calculated area density is 2.06 nm^2 per molecule.

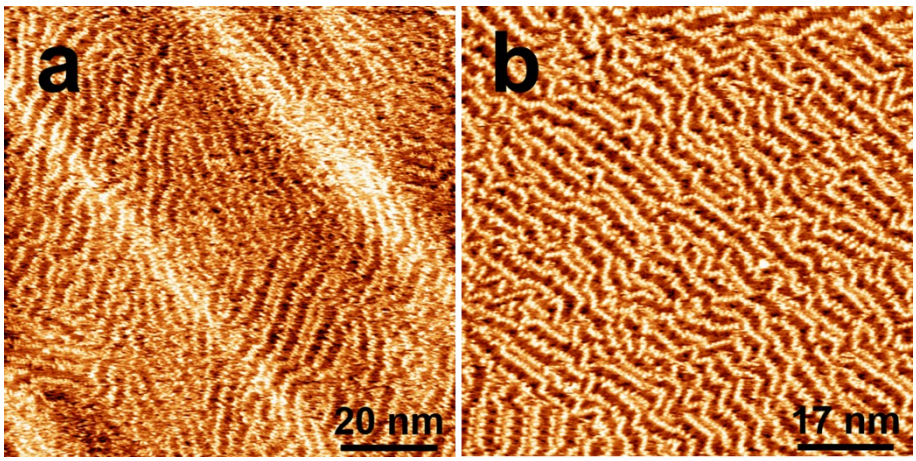

Fig. S4 Large-scale STM images for F-$C_{14}C_7$ at the gas/HOPG interface, showing the disordered phase. Concentration: $C_0 = 1.5 \times 10^{-6}$ M. (a) STM image which is obtained spontaneously after a drop of solution was deposited onto the HOPG surface. (b) STM image which is obtained more than six hours later. Scanning conditions: $I_t = 660$ pA, $V_{bias} = 790$ mV for (a), $I_t = 650$ pA, $V_{bias} = 780$ mV for (b).

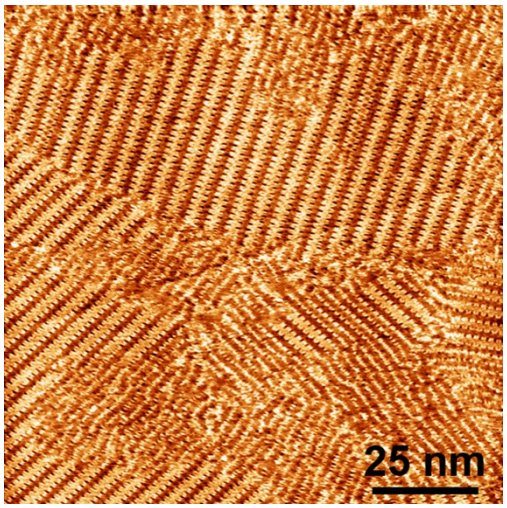

Fig. S5 Large-scale STM image for F-$C_{14}C_{14}$ at the gas/HOPG interface, showing the co-existence of ordered Plier-like structure and random phase. Concentration: C_0 = 1.4 × 10^{-6} M. Scanning conditions: I_t = 630 pA, V_{bias} = 810 mV.

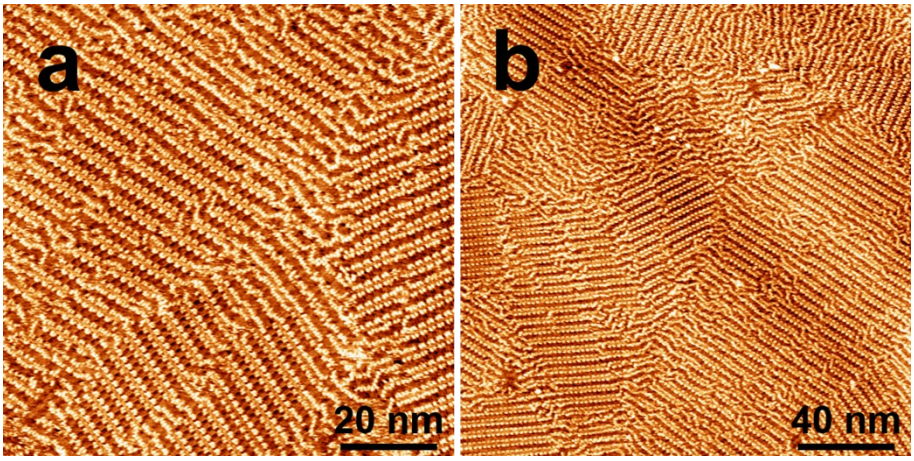

Fig. S6 Large-scale STM images for F-$C_{14}C_7$ at the gas/HOPG interface, showing the co-existence of ordered Plier-like structure and random phase. Concentration: 1/3 C_0. (a) STM image which is obtained spontaneously after a drop of solution was deposited onto the HOPG surface. (b) STM image which is obtained more than six hours later. Scanning conditions: I_t = 630 pA, V_{bias} = 750 mV for (a), I_t = 650 pA, V_{bias} = 750 mV for (b).

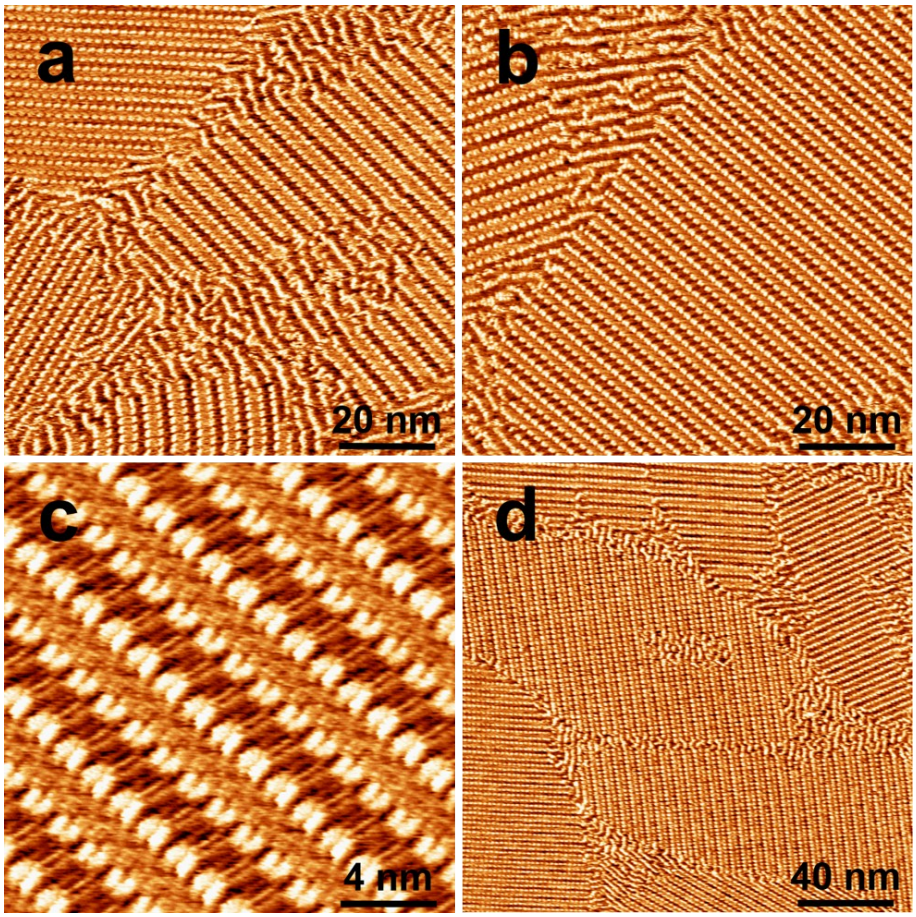

Fig. S7 (a and b) Large-scale STM images for F-$C_{14}C_7$ at the gas/HOPG interface, showing ordered Plier-like structure together with small random areas. Concentration: 1/9 C_0. (c) high-resolution STM image for F-$C_{14}C_7$ at the gas/HOPG interface. (d) STM image which is obtained under a further diluted concentration. Concentration: 1/12 C_0. Scanning conditions: I_t = 600 pA, V_{bias} = 740 mV for (a, b and c), I_t = 710 pA, V_{bias} = 790 mV for (d).

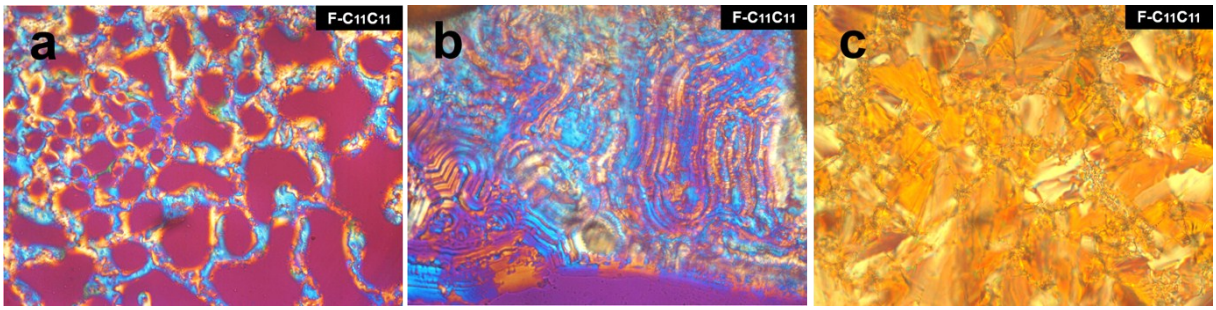

Fig. S8 POM images on the trace of cooling for F-$C_{11}C_{11}$. (a and b) Liquid crystal phases of schlieren and spherulitic textures. (c) Crystalline phase. All of these images are 500× magnified.

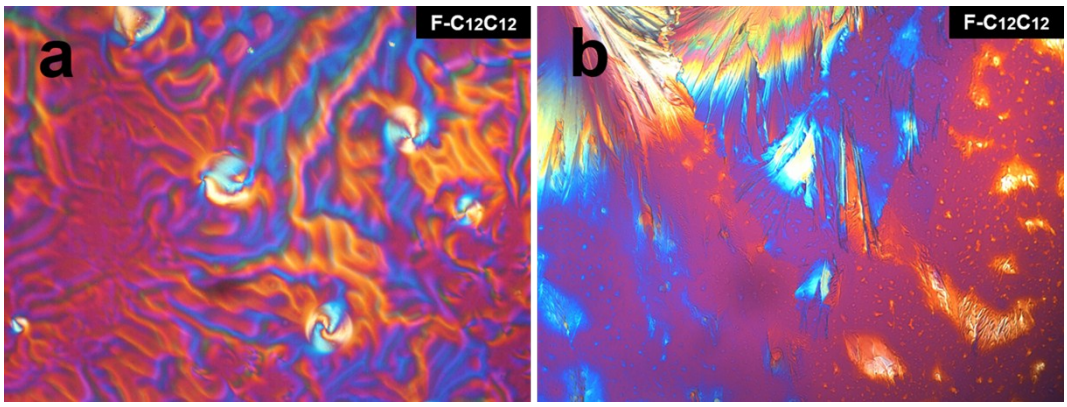

Fig. S9 POM images on the trace of cooling for F-$C_{12}C_{12}$. (a) Liquid crystal phase of schlieren texture. (b) Crystalline phase. All of these images are 500× magnified.

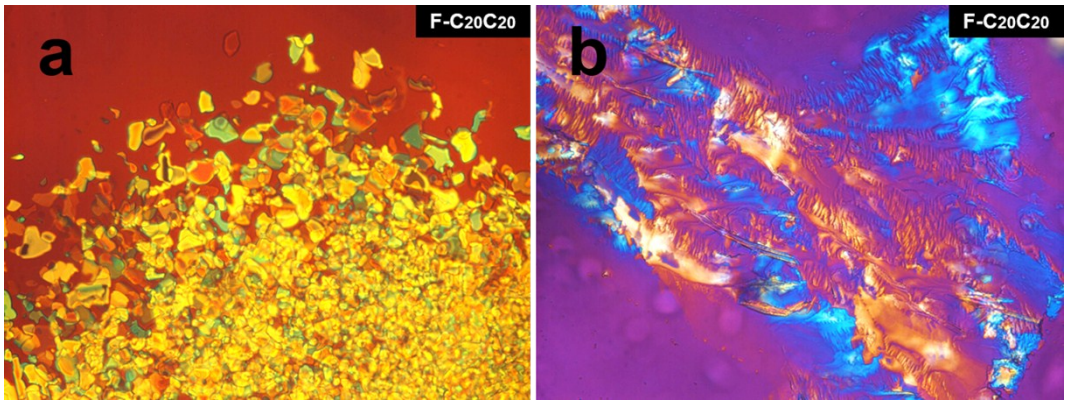

Fig. S10 POM images on the trace of cooling for F-$C_{20}C_{20}$. (a) Liquid crystal phase of spherulitic texture. (b) Crystalline phase. All of these images are 500× magnified.

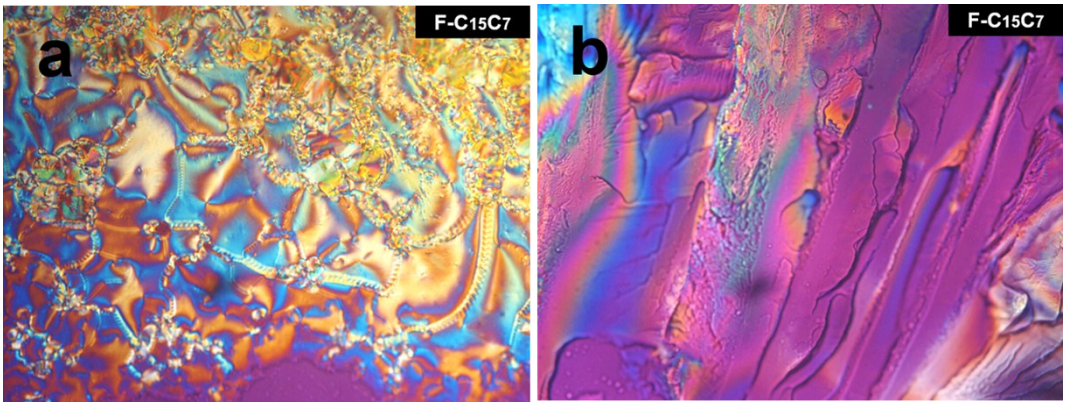

Fig. S11 POM images on the trace of cooling for F-$C_{15}C_{7}$. (a) Liquid crystal phase of schlieren texture. (b) Crystalline phase. All of these images are 500× magnified.