Electronic Supplementary Material (ESI) for ChemComm.

SUPPLEMENTARY INFORMATION

Recrystallization solvent dependent elastic/plastic flexibility of an *n*-dodecyl-substituted tetrachlorophtalimide

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Experimental

Materials

All chemicals and solvents used in the preparation were purchased from Tokyo Kasei Co. and Wako Pure Chemical Industries, Ltd. and used without further purification. 4,5,6,7-Tetrachloro-2-dodecylisoindoline-1,3-dione, **1**, was prepared according to the reported procedure [S1] by heating tetrachlorophthalic anhydride (2.85 g, 10 mmol) and dodecylamine (2.86 g, 10 mmol) in toluene (30 mL) under Dean Stark condition for 12 hours, which was evaporated to dryness under reduced pressure after cooling to ambient temperature. Yiend, 3.1 g (68%). Anal. calc. for $C_{20}H_{25}Cl_4NO_2$: C, 53.00; H, 5.56; N, 3.09; Found: C, 53.29; H, 5.48; N, 3.30 %. ^{1}H NMR (500 MHz, DMSO-d_6): δ 0.81 (d, 3H, CH3), 1.19 (m, 18H, CH2), 1.60 (m, 2H, CH2), 3.62 (t, 2H, CH2). The white solid (30 mg) was then dissolved in 50 mL of dichloromethane or tetrahydrofuran and evaporated slowly under ambient temperature to produce crystals, $\mathbf{1}^{\mathbf{DCM}}$ or $\mathbf{1}^{\mathbf{THF}}$, respectively, suitable for a structure determination.

Physical measurements

Elemental analyses (C, H, N) were carried out on a J-SCIENCE LAB JM10 analyzer at the Instrumental Analysis Centre of Kumamoto University. Single-crystals for X-ray diffraction data of $\mathbf{1}^{\mathbf{DCM}}$ and $\mathbf{1}^{\mathbf{THF}}$ were obtained at 100 K. Their crystal structures were also measured at 300 K, but all data were exactly the same at lower temperatures. The data were collected with a Rigaku XtaLAB mini II diffractometer. The structures were solved by direct methods (SHELXT [S2]) and refined by full-matrix least-squares refinement using the SHELXL [S3] program. Hydrogen atoms were refined geometrically using a riding model. Crystallographic data for $\mathbf{1}^{\mathbf{DCM}}$ and $\mathbf{1}^{\mathbf{THF}}$ are summarized in Table S1. Scanning electron microscopy (SEM) images were collected on a HITACHI SU-8010. Nanoindentation tests were carried out using a Bruker's Hysitron TI Premier Nanomechanical Test Instrument equipped with a diamond Berkovich tip. The (010) face of $\mathbf{1}^{\mathbf{DCM}}$ and (001) face of $\mathbf{1}^{\mathbf{THF}}$ were indented by the load-controlled mode under the conditions of loading for 5s, holding for 2s and unloading for 5s in all measurements.

Table S1. Crystallographic data of $\mathbf{1}^{DCM}$ and $\mathbf{1}^{THF}$.

Compound	$\mathbf{1}^{DCM}$	$\mathbf{1}^{THF}$
formula	$C_{20}H_{25}Cl_4NO_2$	$C_{18}H_{21}Cl_4NO_2$
formula weight	453.21	453.21
crystal system	monoclinic	triclinic
space group	$P2_1/c$	P-1
a / Å	4.9756(2)	4.9963(4)
b / Å	7.2308(2)	5.9993(4)
c / Å	57.2041(19)	36.274(2)
α / º	115.025(4)	90.695(5)
β / º	90	90.006(6)
γ / º	92.612(3)	105.259(6)
V / Å^3	2056.28(12)	1048.87(13)
Z	4	2
T / K	100 K	100 K
R_1	0.0629	0.0875
R_1 (all data)	0.0715	0.0945
wR_2	0.1278	0.2455
wR_2 (all data)	0.1301	0.2482
G.O.F.	1.050	1.282
CCDC	2144528	2144529

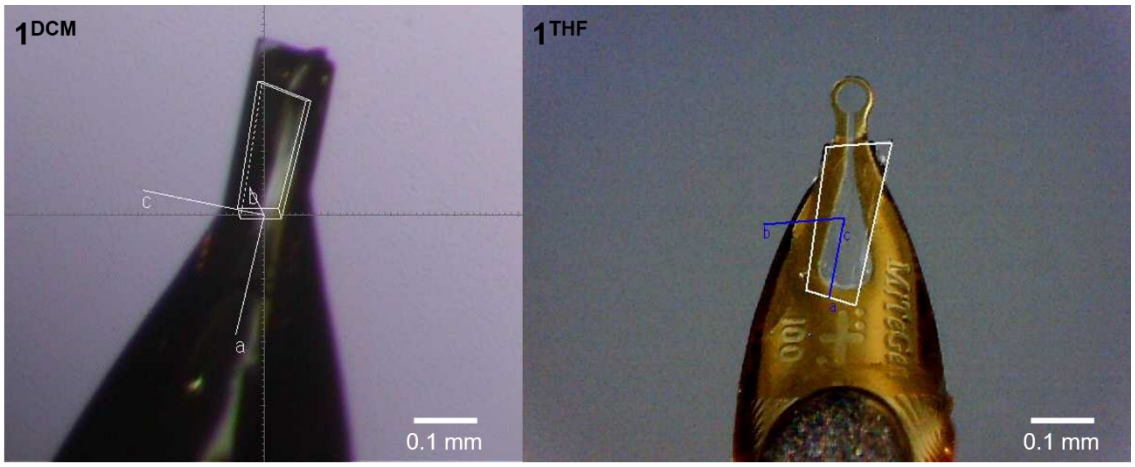


Fig. S1 Face indexing of $\mathbf{1}^{DCM}$ and $\mathbf{1}^{THF}$.

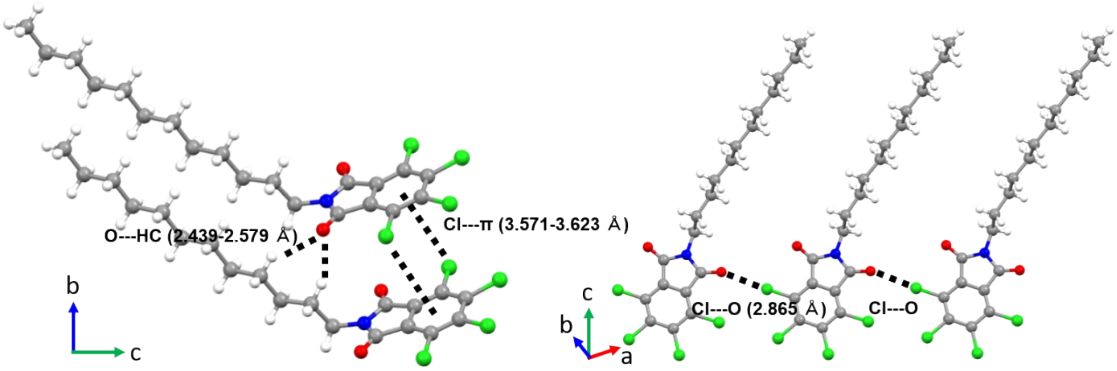


Fig. S2 Intermolecular interactions of $\mathbf{1}^{THF}$.

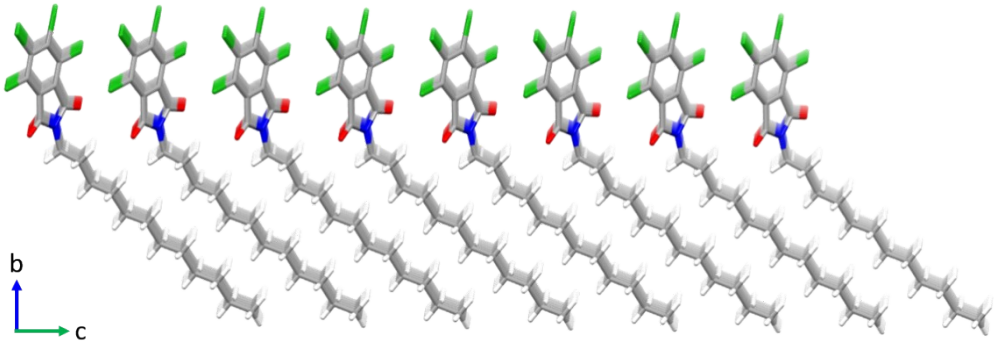


Fig. S3 Formation of 2D sheet-like structure viewed down *a*-axis.

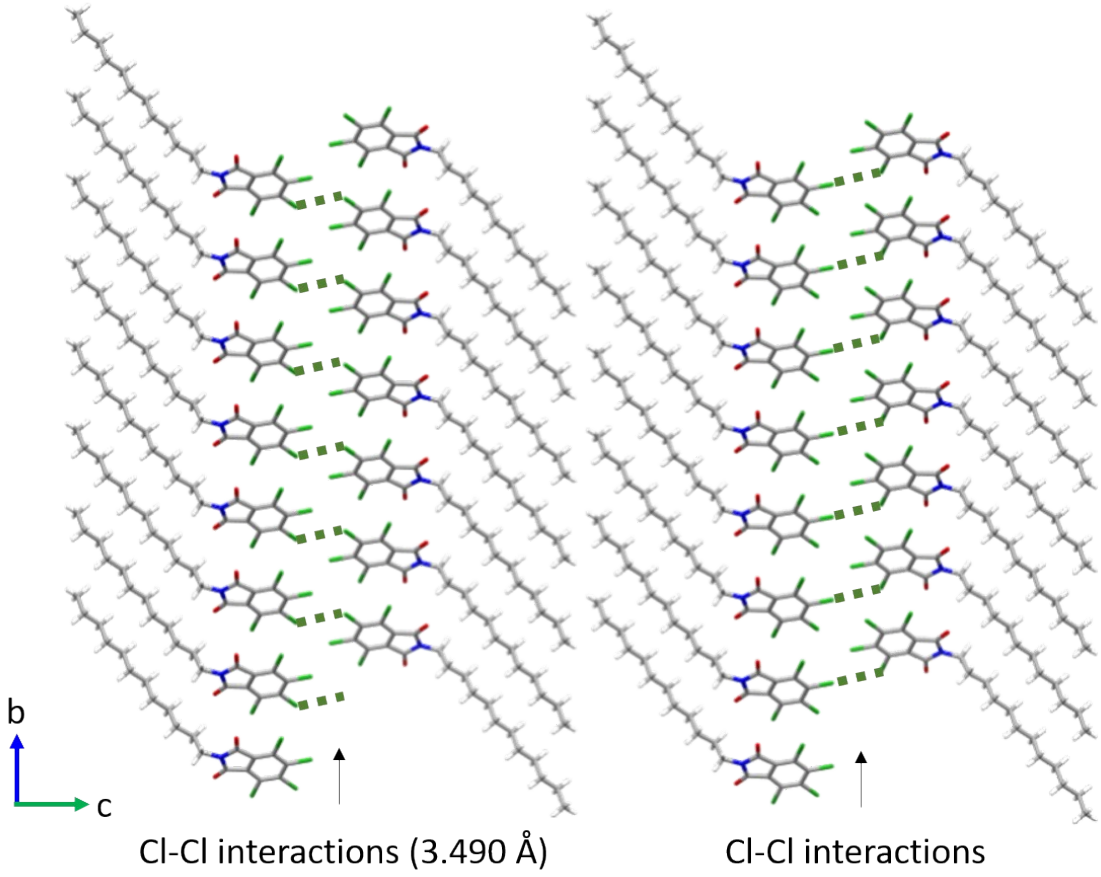


Fig. S4 2D-like structures of $\mathbf{1}^{THF}$ are stitched with Cl---Cl interactions.

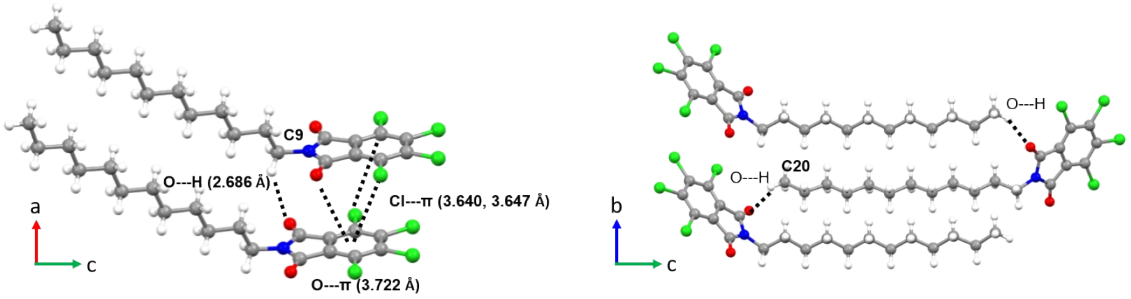


Fig. S5 Intermolecular interactions of $\mathbf{1}^{DCM}$.

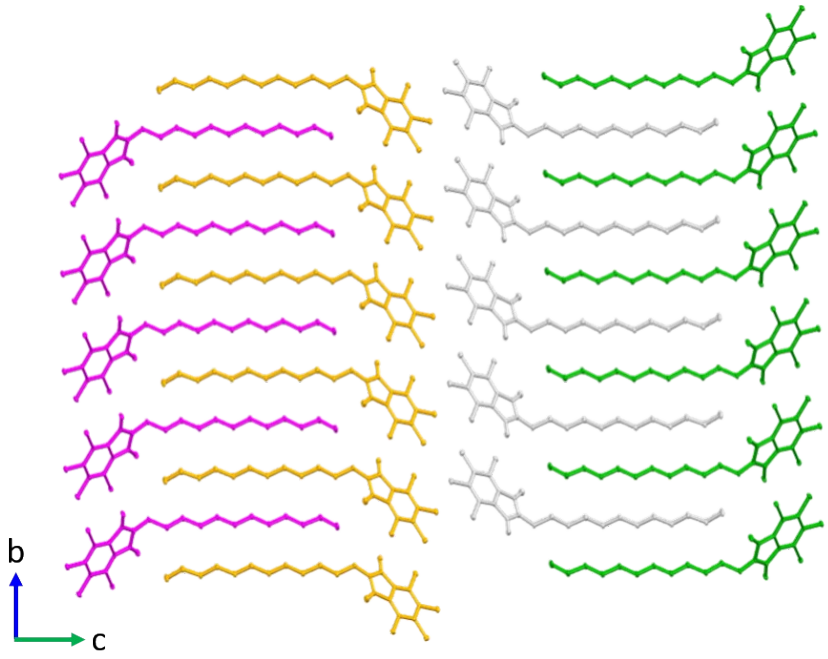


Fig. S6 Packing structure of $\mathbf{1}^{\mathbf{DCM}}$ viewed down *a*-axis with symmetry operation.

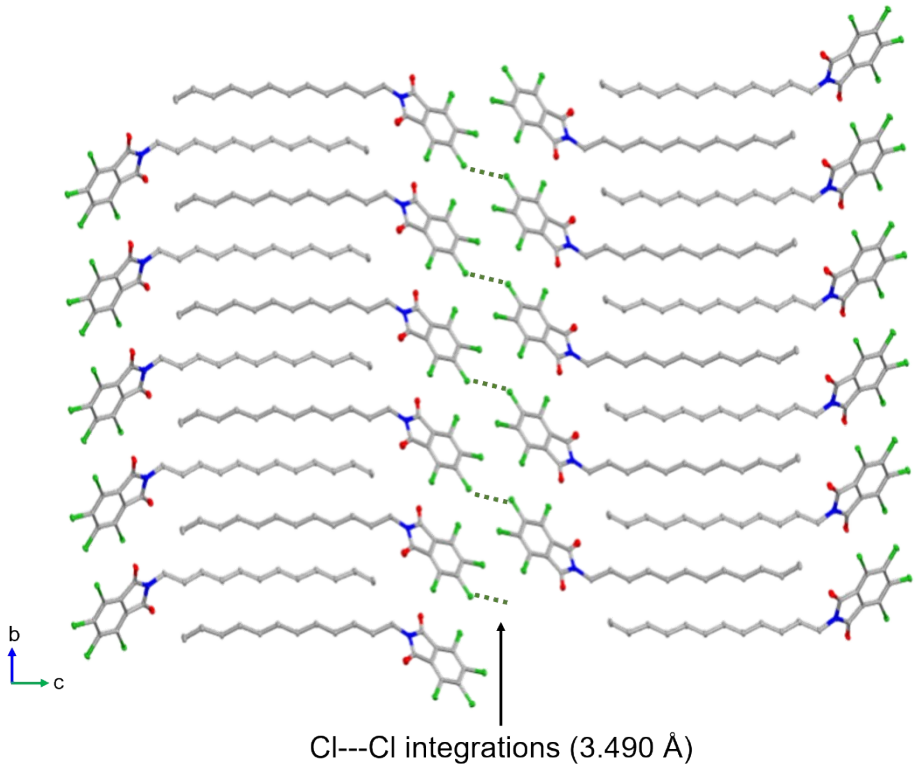


Fig. S7 2D-like structures of $\mathbf{1}^{\mathbf{DCM}}$ are stitched with Cl---Cl interactions.

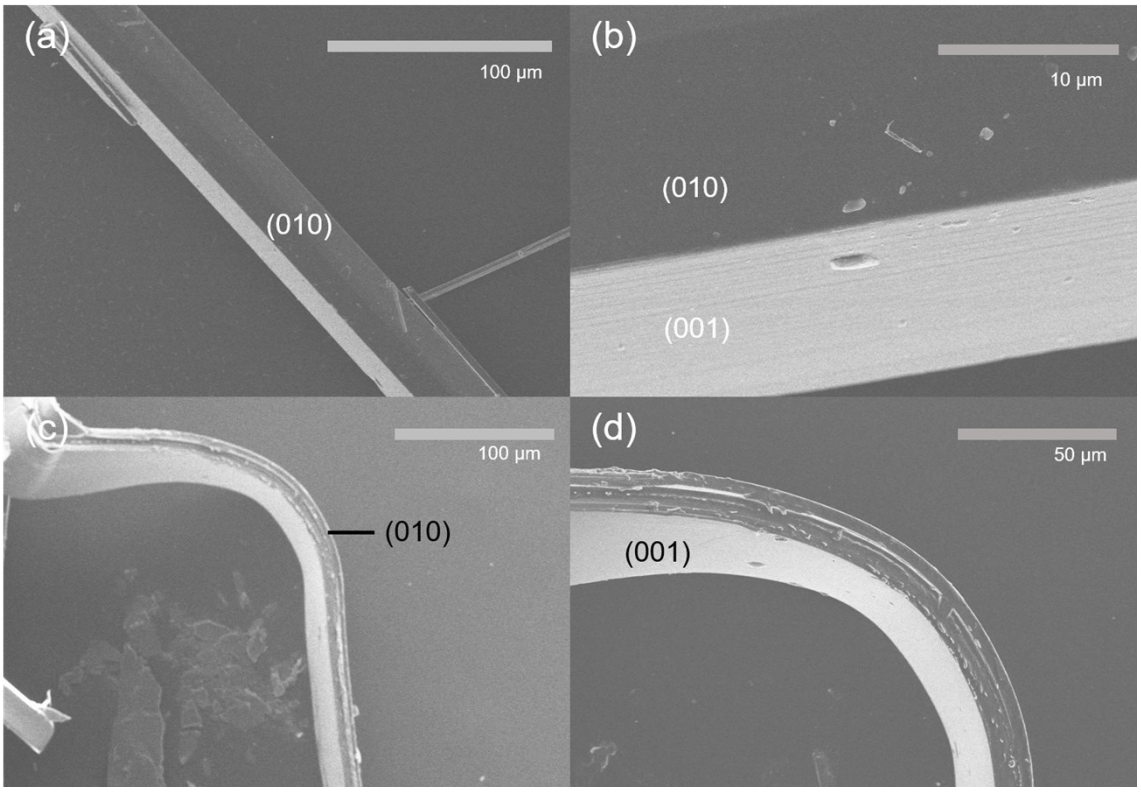


Fig. S8 Scanning electron microscope (SEM) images of $\mathbf{1}^{\mathbf{DCM}}$ (a, b) and $\mathbf{1}^{\mathbf{THF}}$ (c, d).

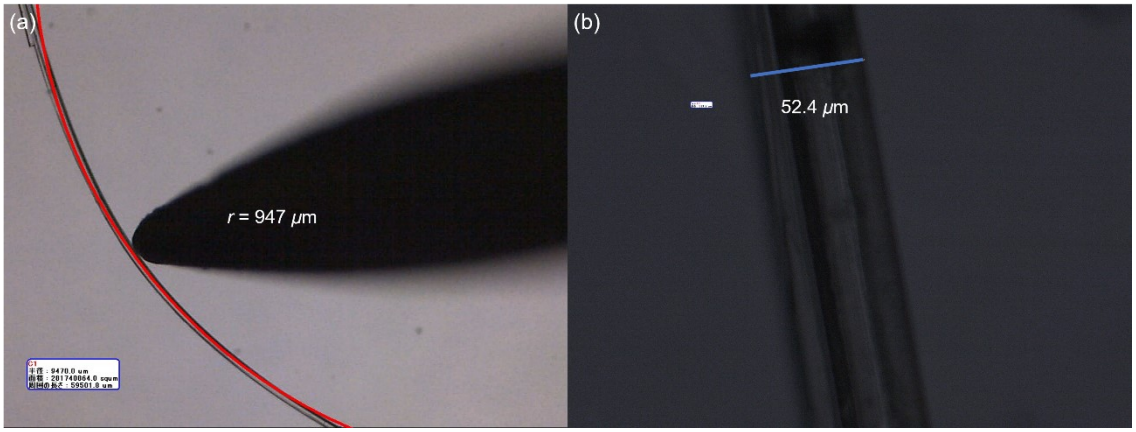


Fig. S9. Bending experiment of $\mathbf{1}^{\mathbf{DCM}}$ (images (a) magnified 40 times, image (b) magnified 400 times) having ε = 2.8%. Grease was partially applied to prevent the slip of crystals from the glass surface before applying mechanical forces. The bending strain (ε) was estimated using the Euler-Bernoulli equation [S4] $\varepsilon = (t/2R)$, where t is the thickness of the crystal (52.4 μm) and R is the Radius of the curvature (947 μm).

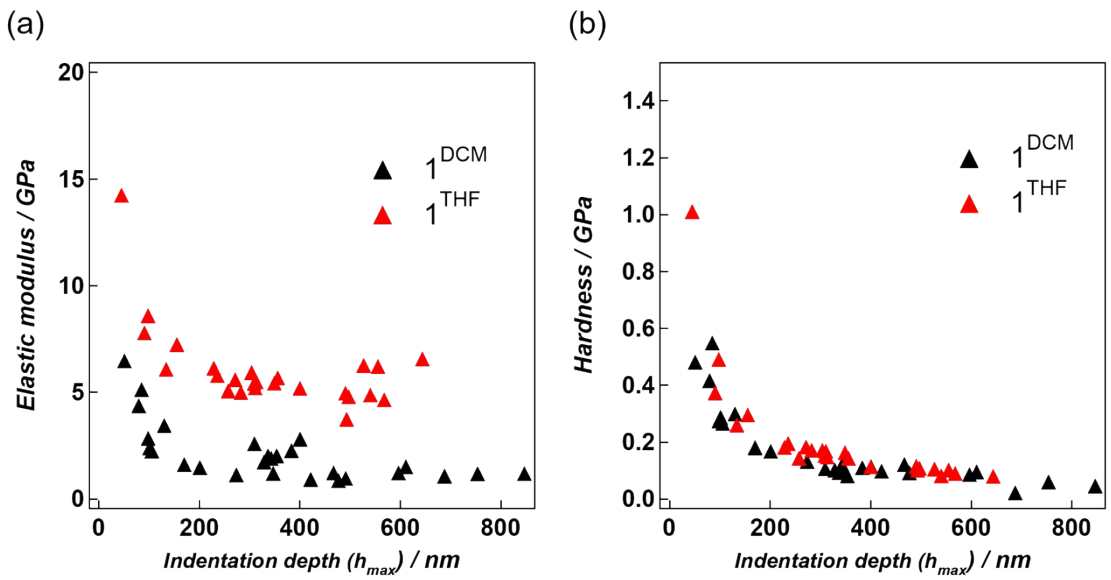


Fig. S10 Indentation depth (h_{max}) depending on Elastic modulus (GPa) and Hardness (GPa) for **1**^{DCM} and **1**^{THF}.

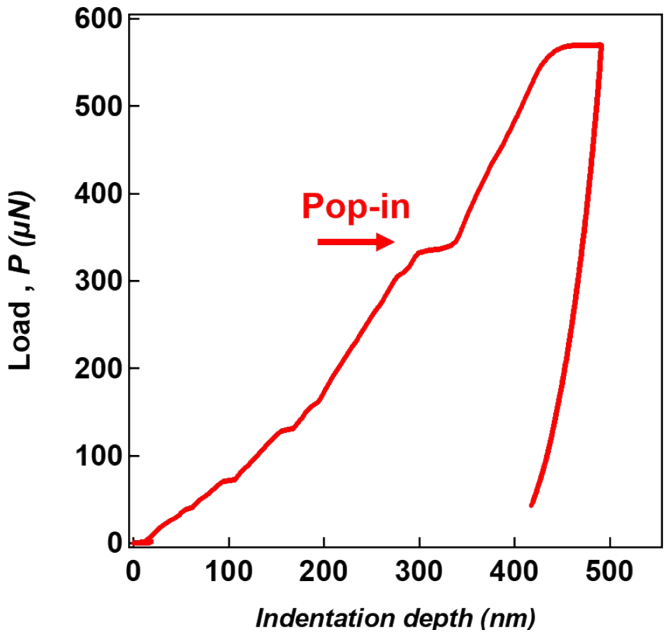
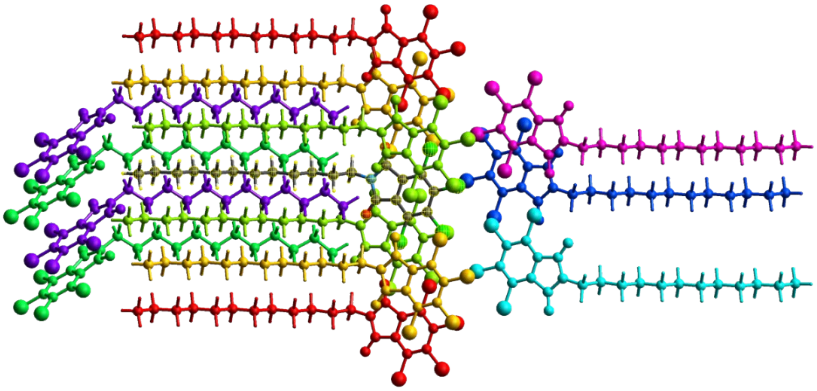
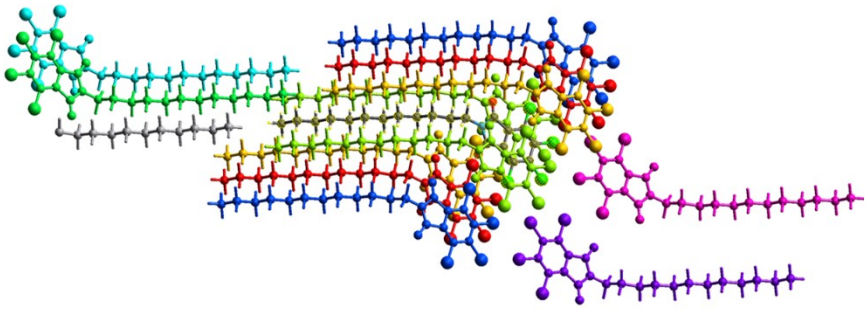


Fig. S11 *P-h* curve of **1**^{THF} when the maximum loading 570 μN was applied on the (001) face.



	N	Symop	R	Electron Density	E_ele	E_pol	E_dis	E_rep	E_tot	
	2	x, y, z	8.78	HF/3-21G	−7.5	−1.6	−11.1	14.1	−7.2	
	2	x, y, z	7.23	HF/3-21G	−6.4	−0.8	−22.8	11.7	−18.1	
	2	x, y, z	4.98	HF/3-21G	−18.5	−3.6	−79.6	46.3	−55.3	Based on Cl---π, CO---π and CH---O
	2	−x, y+1/2, −z +1/2	15.46	HF/3-21G	−5.8	−2.6	−49.3	23.3	−33.1	
	1	−x, −y, −z	16.37	HF/3-21G	−4.0	−0.1	−10.1	12.7	−2.9	
	1	−x, −y, −z	14.29	HF/3-21G	−6.3	−0.3	−23.3	16.9	−13.8	Based on Cl---Cl
	2	−x, y+1/2, −z +1/2	14.94	HF/3-21G	−4.2	−0.9	−57.6	22.5	−38.5	
	1	−x, −y, −z	15.80	HF/3-21G	−3.0	−0.1	−9.8	10.5	−3.5	

Figure S12 The estimated interaction energies of $\mathbf{1}^{DCM}$ with various molecular dimers using CE-B3LYP method.



	N	Symop	R	Electron Density	E_ele	E_pol	E_dis	E_rep	E_tot	
	2	x, y, z	6.00	HF/3-21G	−20.4	−4.6	−82.8	48.5	−59.1	
	2	x, y, z	6.72	HF/3-21G	0.1	−0.4	−30.1	7.8	−20.9	Based on Cl---π, CO---π and CH---O
	2	x, y, z	5.00	HF/3-21G	−20.2	−3.5	−80.6	48.7	−56.0	
	1	−x, −y, −z	27.51	HF/3-21G	−1.1	−0.0	−11.6	5.7	−7.0	
	1	−x, −y, −z	27.55	HF/3-21G	−0.9	−0.0	−11.1	4.9	−6.9	
	2	x, y, z	8.76	HF/3-21G	−8.0	−1.8	−12.1	15.3	−7.9	Based on CH---HC or Cl---Cl
	1	−x, −y, −z	14.57	HF/3-21G	−3.5	−0.1	−9.8	10.6	−3.9	
	1	−x, −y, −z	16.50	HF/3-21G	−3.1	−0.1	−9.6	10.1	−3.6	

Figure S13 The estimated interaction energies of $\mathbf{1}^{THF}$ with various molecular dimers using CE-B3LYP method.

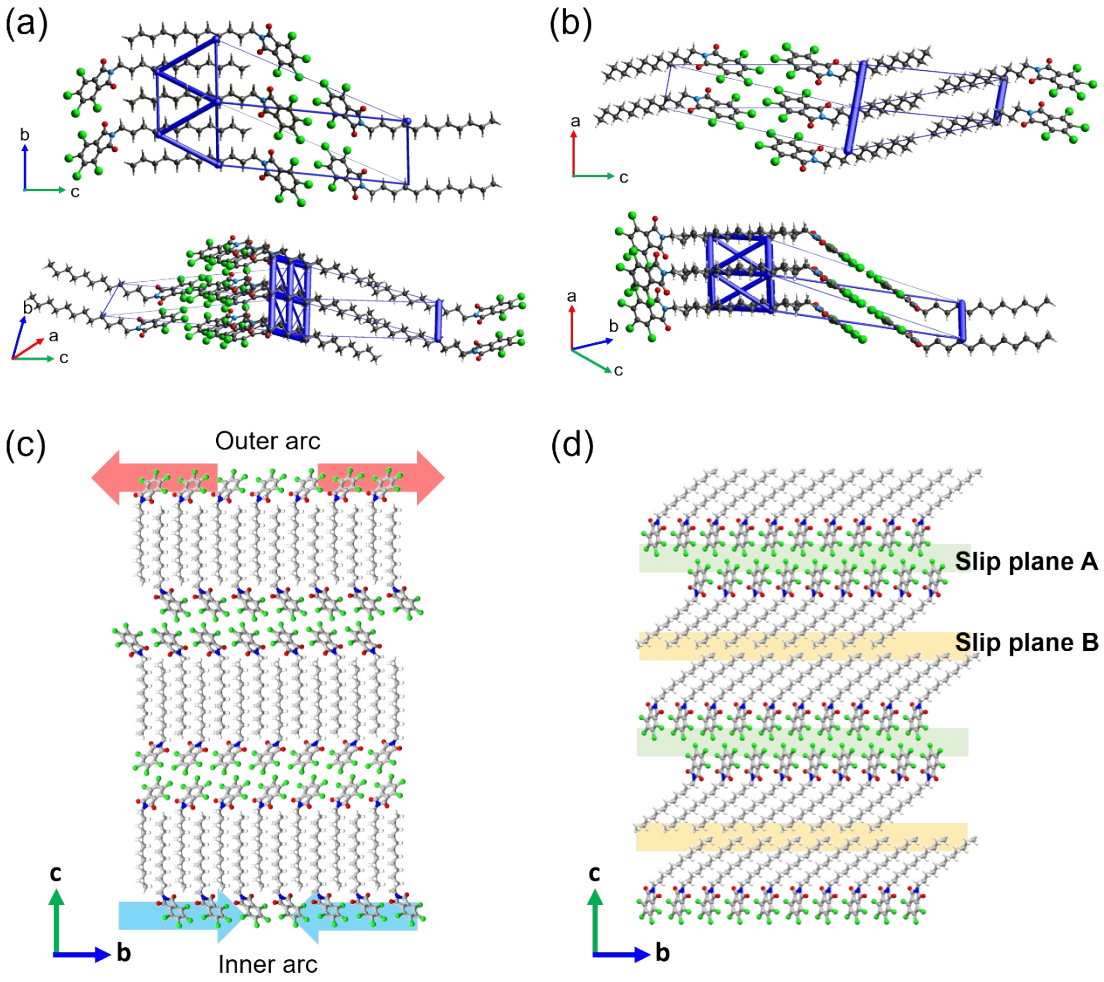


Fig. S14 Energy framework analyses of (a) $\mathbf{1}^{DCM}$ and (b) $\mathbf{1}^{THF}$. Mechanism for (c) elastic bending ($\mathbf{1}^{DCM}$) and (d) plastic bending ($\mathbf{1}^{THF}$).

References

[S1] T. Borowiak, I. Wolska, B. Brycki, A. Zieliński, I. Kowalczyk, *J. Mol. Struct.*, 2007, **833**, 197–202.

[S2] G. M. Sheldrick, Acta Cryst. 2015, A**71**, 3-8.

[S3] G. M. Sheldrick, Acta Cryst. 2015, C**71**, 3-8.

[S4] S. Timoshenko, Strength of materials; Van Nostrand Company: New York, 1940.