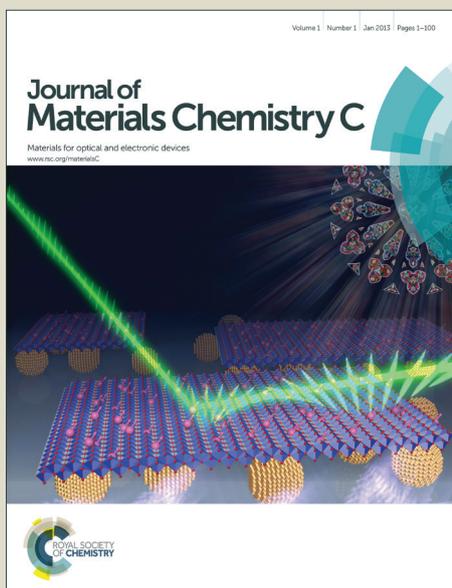


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ARTICLE

Enhanced performance in isoindigo based organic small molecule field-effect transistors through solvent additives

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Isoindigo based small molecules have attracted much attention in the field of optoelectronic devices due to their broad absorbance and high charge carrier mobilities. Herein, we have investigated the field-effect transistor (FET) characteristics of a series of isoindigo based donor-acceptor-donor (D-A-D) small molecules containing one, two or three thiophene moieties (named IDT, ID2T, and ID3T, respectively) or an ethylenedioxythiophene moiety (IDED). In order to improve the carrier mobility, 1-chloronaphthalene (CN) and 1,8-diodooctane (DIO) as solvent additives were used. Films were processed with various concentrations of solvent additives and their morphology was examined using atomic force microscopy (AFM). This study provides an understanding of the influence of volatile solvent additives and processing conditions on the morphology of small molecule bulk heterojunction systems, and serves as a guide to aid in the use of solvent additives in small molecules FET devices.

Introduction

Solution processing fabrication is a promising technique for producing flexible, large-area and low-cost printable electronic devices.¹⁻⁵ Solution-processable small molecules have recently attracted considerable attention due to their well-defined chemical structures, simple synthesis, good crystallinity and high purity compared to polymers.³⁻⁸ In recent years, solution-processable small molecule organic field-effect transistors (OFETs) with mobilities above $10 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ have been reported using inkjet printing and solution-shearing.⁹⁻¹¹ Many researchers continue to pursue methods to improve carrier mobilities through the development of deposition methods, the synthesis of the novel materials, the study of interfaces and so on.¹⁰

Isoindigo ((*E*)-1*H*,1'*H*-[3,3']biindolylidene-2,2'-dione) is an isomer of the well-known dye indigo; both dye topologies are defined by two γ -lactam rings linked by a double bond.¹³⁻¹⁷ The isoindigo moiety has received considerable attention as building block for solution-processable organic semiconductors. In 2010, Reynolds *et al.* first reported solution-processed organic photovoltaic devices (OPVs) using isoindigo-based donor-acceptor-donor (D-A-D) and acceptor-donor-acceptor (A-D-A) small molecules incorporating isoindigo and bithiophene moieties. In 2012, a hole mobility of $3.62 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ was achieved by Pei and coworkers in OFETs using an isoindigo-based polymer designed using branching point

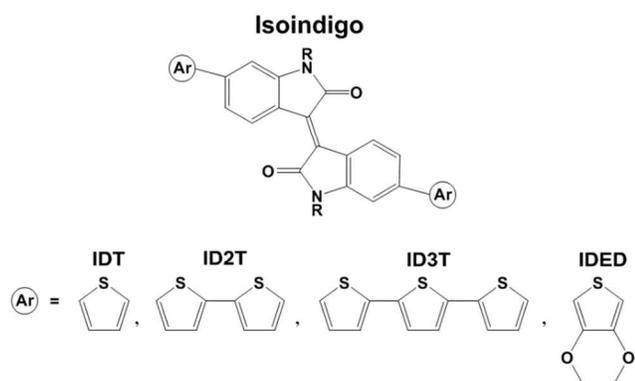
variation strategy.¹⁸ In these reports, the electron-withdrawing character and high crystallinity of the isoindigo moiety was leveraged to yield symmetric D-A-D materials with planar structures and excellent π -conjugation.¹⁹⁻²⁰

Although they possess promising photophysical properties, isoindigo based small molecule OFETs currently exhibit relatively low mobilities ($\sim 0.1 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$) compared to isoindigo based conjugated polymers ($\sim 4 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$).^{13-16, 21,22} Recently, it has been noted that certain hydrogen-terminated oligothiophenes donors provided advantages in solar cell devices relative to analogous alkyl chain terminated molecules, however, hydrogen-terminated isoindigo molecules have not yet been thoroughly investigated in OFET devices.

In order to improve device performance, solvent additive processing has extensively been used as a method to improve solar cell performance and has recently been introduced as an effective method to process other optoelectronic devices such as FETs as well.²³ For instance, in 2013, Seo and coworkers reported the use of processing additives to modify the electronic properties, charge transport behavior and molecular self-assembly of small-molecule based OPVs.²⁴ In 2014, Li *et al.* demonstrated improved performance in solution-processed 6,13-bis(triisopropylsilyl)ethynylpentacene (TIPS-pentacene) FETs blended with a series of small molecule additives (4-butylbenzoic acid (BBA), 4-hexylbenzoic acid (HBA) and 4-octylbenzoic acid (OBA)), which have similar structures with same hydrophilic head but different length of hydrophobic

tail.²⁵ From these reports, it is apparent that solvent additives may

Scheme 1 Chemical structures of isoindigo based donor-acceptor-donor (D-A-D)



small molecules. (R = ethylhexyl)

comprise a variety of high-boiling liquids, and that appropriate amounts of these additives are able to induce the formation of well-ordered crystalline domains and improved film morphology, resulting in enhanced device performance.

In this work, we have studied a series of hydrogen terminated, isoindigo based D-A-D small molecules containing a variable number of thiophene or ethylenedioxythiophene (EDOT) moieties.²⁶ Optical absorption, ultraviolet photoelectron spectroscopy (UPS) and density functional theory (DFT) calculations reveal significant differences in the optical and electronic characteristics of the materials. Characterization of morphology by atomic force microscopy (AFM) and carrier mobilities (using OFET devices) reveals that solvent additive processing is an effective way to influence film microstructure and improve OFET performance.

Results and discussion

Molecular structures

Scheme 1 shows the chemical structures of the isoindigo based D-A-D small molecules used in this study. Each molecule in the series consists of an isoindigo core, which serves as an electron accepting group, with flanking oligothiophenes of variable length, which serve both as electron donating groups and π -bridges in this topology. The compounds comprising mono-, bi- and ter-thiophene are abbreviated IDT, ID2T and ID3T, respectively. A fourth EDOT containing molecule is abbreviated IDED. Differences in the flanking moieties affect the conjugation length and electron donating strength of each molecule. Generally, increasing the number of thiophene moieties increases conjugation length of small molecules while also affecting solubility and π - π stacking in the solid state.²⁷ Additionally, the electron donating strength of different donor units influences the frontier orbital energy levels and intramolecular charge transfer (ICT) bands as well.²⁸⁻
³⁰ The energy band structures of IDT, ID2T, ID3T and IDED were studied via UV-vis absorption spectroscopy, UPS and DFT (see Fig. S3-S5). These results are summarized in Table 1.

Characteristics of OFETs

OFETs based the series of isoindigo small molecules (IDT, ID2T, ID3T and IDED) were fabricated with various concentrations of DIO and CN additives (none, 0.2%, 0.5% and 1%). Figure 1 shows the transfer characteristics of IDT, ID2T,

Table 1 Energy levels of isoindigo small molecules

Materials	$E_{\text{HOMO}}^{\text{a}}$ (eV)	$E_{\text{LUMO}}^{\text{b}}$ (eV)	E_{g}^{c} (eV)	$E_{\text{HOMO}}^{\text{d}}$ (eV)	$E_{\text{LUMO}}^{\text{e}}$ (eV)	E_{g}^{f} (eV)
IDT	-5.37	-2.87	2.50	-6.15	-4.41	1.74
ID2T	-5.15	-2.90	2.25	-5.70	-3.98	1.73
ID3T	-5.00	-2.92	2.08	-5.52	-3.86	1.66
IDED	-5.06	-2.65	2.41	-6.03	-4.23	1.80

^{a, b, c} Calculated by DFT. ^d Measured by UPS. ^e Calculated from ^d + ^f. ^f Measured by UV-vis absorption spectrum.

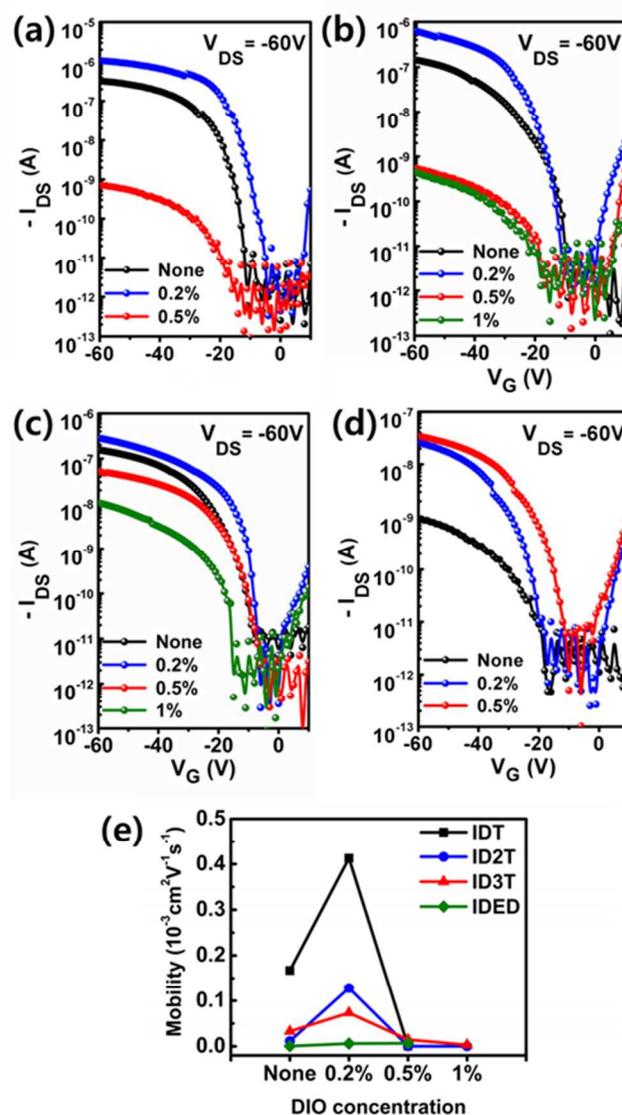


Fig. 1 Transfer characteristics of (a) IDT, (b) ID2T, (c) ID3T, (d) IDED FETs processed with various concentrations of DIO and (e) DIO concentrations versus mobilities corresponding to isoindigo small molecule FETs.

ID3T and IDED FETs processed with 0.2%, 0.5% and 1% DIO (See Fig. S1 for output characteristics). OFETs with pristine IDT, ID2T, ID3T and IDED exhibited hole mobilities of 1.67×10^{-3} , 1.15×10^{-4} , 3.28×10^{-4} and 4.57×10^{-6} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$, respectively. The performance of IDT FETs increased slightly upon adding 0.2% DIO (4.13×10^{-3} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$), while the IDT FET with 0.5% DIO exhibited a decreased hole mobility of 3.67×10^{-6} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$. The ID2T FETs show a strong dependence in performance with the use of DIO. Examination of the transfer curves (Fig. 1(b)) reveals increased source-drain current (I_{DS}) for ID2T FETs processed with 0.2% DIO, but decreased I_{DS} for devices processed with high loadings of 0.5% and 1% DIO. The observed hole mobilities were 1.28×10^{-3} (for 0.2% DIO), 1.58×10^{-6} (for 0.5% DIO) and 8.31×10^{-7} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$ (for 1% DIO). Again, the highest hole mobility for ID3T (7.34×10^{-4} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$) was obtained using 0.2% DIO, while increasing concentrations led to a decrease in current. A slight improvement was observed for IDED FETs processed with 0.5% DIO leading to increased I_{DS} at high gate bias, however, high loading of DIO introduced difficulties in device fabrication due to dewetting from substrates. Fig. 1(e) provides a summary of the mobilities obtained using various DIO concentrations.

The HOMO levels of the small molecules lie at relatively low energies relative to the Au source and drain electrodes, indicating that charge injection barriers may limit current through the devices and result in artificially low mobilities (Figure S6, Supporting Information). IDED in particular, exhibits the deepest HOMO value of - 6.15 eV with a hole injection barrier of 1.29 eV and shows an optimal hole mobility of only 9.77×10^{-5} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$. However, the HOMO value (6.03 eV and charge injection barrier (1.17 eV) for IDT are similar to those of IDED, while the observed mobilities (up to 1.10×10^{-2} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$) are among the highest out of the series, suggesting that charge injection barriers have a minor influence on the measured carrier mobilities of this series compared to other material properties such as film morphology.

Increasing the concentration of DIO above 0.2% led to a decrease in device performance. This decrease in mobility is consistent with a decrease in crystallinity as observed in X-ray diffraction (XRD) patterns (Figure S7, Supporting Information) and an increase in charge trapping as evidenced by the increased absolute threshold voltage. This decrease in device performance mirrors similar sharp drops in solar cell performance which are observed when excessive additive is used in small molecule solar cell devices.³¹

To investigate the effect of other additives on electronic transport in isoindigo based small molecule FETs, CN was chosen as a processing additive which has been successfully used in OPVs. All devices were fabricated using the same procedure used with DIO involving various CN concentrations in a solvent of chloroform. Figure 2 shows the transfer

characteristics of IDT, ID2T, ID3T and IDED FETs processed with 0.2%, 0.5% and 1% CN. (See Fig. S2 for output characteristics). It is noteworthy that device performance for isoindigo small molecule FETs show significant differences upon incorporation of CN additive compared to DIO. In contrast to DIO, 0.5% CN enhances the device performance of IDT FETs, leading to an increase in hole mobility from 1.67×10^{-3} to 1.10×10^{-2} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$. There was no increase in device performance for ID2T FETs processed with various CN concentrations, indicating that the additive CN is not suitable for ID2T. A large improvement in I_{DS} was observed for ID3T FETs processed with the 0.2% CN (to 1.37×10^{-2} from 3.28×10^{-4} for no additive). In contrast to DIO, IDED FETs exhibited an increased mobility (9.77×10^{-5} for 1% CN) of almost two orders of magnitude compared to the control device (4.57×10^{-6} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$ for no additive). Figure 2(e) displays mobilities versus CN concentrations added in isoindigo based small molecule FETs. All device parameters are summarized in Table 2.

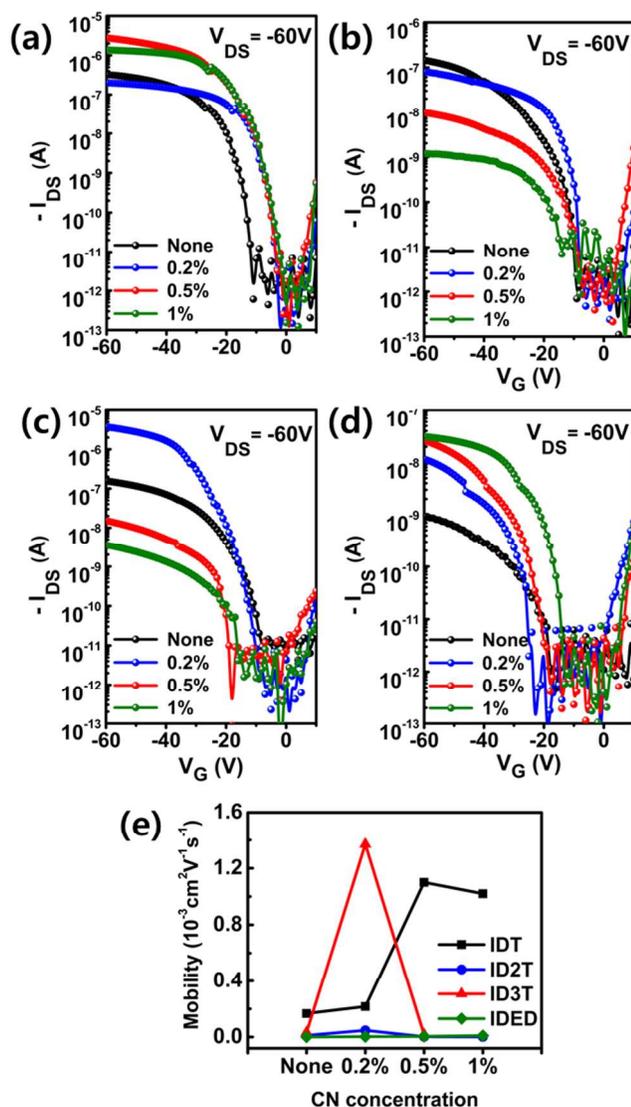


Fig. 2 Transfer characteristics of (a) IDT, (b) ID2T, (c) ID3T, (d) IDED FETs prepared with various concentrations of CN and (e) CN concentrations versus mobilities corresponding to isoindigo small molecules.

It is noteworthy that the threshold voltage varies somewhat upon adding different concentrations of solvent additives. In particular, large quantities of DIO appear to increase the absolute threshold voltage required to turn the device on. Since the threshold voltage indicates the gate potential required to form an accumulation layer of mobile charge carriers in the transport channel, this shift in threshold voltage indicates that excessive amounts of DIO may introduce trap sites for holes, or otherwise reduce the number of mobile in the film. The use of CN shows a similar general trend, although the effect is smaller with CN than DIO with smaller changes in threshold voltage.

Morphology

In order to investigate how solvent additives influence device performance, surface morphologies were studied using AFM. Isoindigo based small molecule films were prepared on Si substrates treated with octyltrichlorosilane (OTS). Figures 3 and 4 show the surface topographic images ($2 \mu\text{m} \times 2 \mu\text{m}$) of IDT, ID2T, ID3T and IDED as a function of DIO and CN concentrations, respectively. The IDT film without additive displays a crystalline morphology with root mean square (rms) roughness of 13.4 nm as shown in Fig. 3(a). Interestingly, the morphologies of the IDT films became more crystalline upon increasing DIO concentrations. When 0.5% DIO was added, the IDT film seemed adopted a morphology resembling a needle-like network, and exhibited a surface roughness of 15.9 nm. The IDT film with 1% DIO was not uniformly wetted on the substrate. Pristine ID2T films featured sparsely raised spots with a surface roughness of 2.7 nm. Changes in morphology are observed upon adding DIO as shown in Fig. 3(b). ID2T films with DIO appeared to be more crystalline and had much larger grain sizes compared to pristine ID2T. Surface topographic image of ID3T with no additive reveal small oblong shaped grains. By adding 0.2% DIO, the morphology transitioned into larger grain sizes with rms roughness decreasing from 7.4 to 2.4 nm (Fig. 3(c)), then increasing for higher DIO loading. In the case of the IDED films, the topographic results are similar to those of the ID2T film. The IDED surfaces exhibited large sized grains with increasing DIO concentrations. Overall, these results demonstrate that

Table 2 Device performance of the isoindigo small molecules FETs.

Parameters	Additives	concentrations	IDT	ID2T	ID3T	IDED	
Mobility ($\text{cm}^2\text{V}^{-1}\text{s}^{-1}$)	DIO	None	1.67×10^{-3}	1.15×10^{-4}	3.28×10^{-4}	4.57×10^{-6}	
		0.2%	4.13×10^{-3}	1.28×10^{-3}	7.34×10^{-4}	6.02×10^{-5}	
		0.5%	3.67×10^{-6}	1.58×10^{-6}	1.47×10^{-4}	6.69×10^{-5}	
		1%	-	8.31×10^{-7}	2.99×10^{-5}	-	
		0.2%	2.17×10^{-3}	4.71×10^{-4}	1.37×10^{-2}	2.85×10^{-5}	
		0.5%	1.10×10^{-2}	2.25×10^{-5}	1.73×10^{-4}	4.59×10^{-5}	
	CN	1%	1.02×10^{-2}	9.16×10^{-6}	1.19×10^{-4}	9.77×10^{-5}	
		DIO	None	5.34×10^5	2.09×10^5	1.67×10^4	2.01×10^3
			0.2%	3.19×10^6	6.17×10^5	9.94×10^5	3.70×10^4
			0.5%	1.89×10^3	3.77×10^3	1.76×10^5	6.90×10^4
1%			-	2.23×10^3	2.12×10^4	-	
0.2%			5.96×10^6	1.69×10^5	1.66×10^7	2.17×10^5	
0.5%	1.08×10^7		1.08×10^4	1.70×10^5	6.37×10^4		
CN	1%	8.95×10^6	1.32×10^3	2.25×10^3	5.36×10^4		
	DIO	None	-14.78	-10.71	-11.14	-21.73	
		0.2%	-9.69	-9.62	-8.02	-20.71	
		0.5%	-17.45	-14.06	-9.95	-13.42	
		1%	-	-14.18	-13.92	-	
		0.2%	-9.26	-9.23	-11.10	-23.93	
0.5%		-10.09	-9.57	-19.35	-21.45		
CN	1%	-10.31	-12.03	-15.61	-14.64		

incorporation of appropriate amounts of solvent additives with these isoindigo based small molecules leads to changes in topographic features, including the formation of large crystal grains.

Similar changes in morphology were observed when small amount of CN were used as shown in Fig. 4. The IDT films with CN (rms roughness for 0.2%, 0.5% and 1% are 7.6, 1.6 and 3.4 nm, respectively) form more crystalline morphologies with decreased surface roughness compared to pristine films (rms roughness of 13.4 nm), as shown in Fig. 4(a). The IDT film with 0.5% CN revealed a very low surface roughness of 1.6 nm, which corresponded to optimal device performance. ID2T films with CN exhibited a decrease in mobility, possibly due to excessive grain size in spite of the crystalline formation. The surface morphologies of ID3T films with 0.2% CN featured a decreased roughness of 1.8 nm, where the device performance was dramatically increased. IDED films with CN additives exhibited excessively large sized grains, with low device performance.

It is clear that the addition of DIO or CN as solvent additives changes the morphologies of isoindigo films, resulting in either more crystalline or smaller sized grains. For DIO additive (in the case of ID2T), crystalline morphologies with suitable grain sizes and smooth surfaces lead to enhanced OFET performance, while for CN additive, smaller sized grains increased the device performance (in the case of ID3T) when the films had a smooth morphology. In order to characterize the influence of solvent additives on the crystallinity of the films, XRD patterns were collected for the small molecule films processed with and without additives (Fig. S7, Supporting Information). XRD patterns of ID2T films processed with 0.2%, 0.5% and 1% DIO are shown in Fig. S7(b). An increase in the intensity of the peak at $\sim 6^\circ$ for the ID2T film with 0.2% DIO was observed and the intensity of this feature decreased upon further increasing the DIO concentration. These findings support the change of mobilities in ID2T FETs prepared with DIO. In addition, the ID3T processed with 0.2% CN exhibited strong XRD features at $\sim 7^\circ$ and 14° , which disappeared upon

adding more CN as shown in Fig. S7(c). These results suggest a good correlation between the mobility and crystallinity

Conclusions

We investigated the characteristics of a series of isoindigo based D-A-D small molecules; IDT, ID2T, ID3T and IDED. The four isoindigo small molecules exhibit energy levels which follow the identity of the flanking thiophene oligomers. In particular, the HOMO levels of IDT, ID2T and ID3T (-6.15, -5.70 and -5.52 eV, respectively) were gradually decreased with

an increasing number of thiophene rings, while the HOMO value of IDED was -6.03 eV. These materials were effectively used in OFET devices, where enhanced performance was achieved through solvent additive processing. The hole mobility of IDT was greatly improved using 0.5% CN from 1.67×10^{-3} to 1.10×10^{-2} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$. ID2T FETs exhibited hole mobility increases from 1.15×10^{-4} to 1.28×10^{-3} $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$, when 0.2% DIO was added. The ID3T FETs showed hole mobility increases from

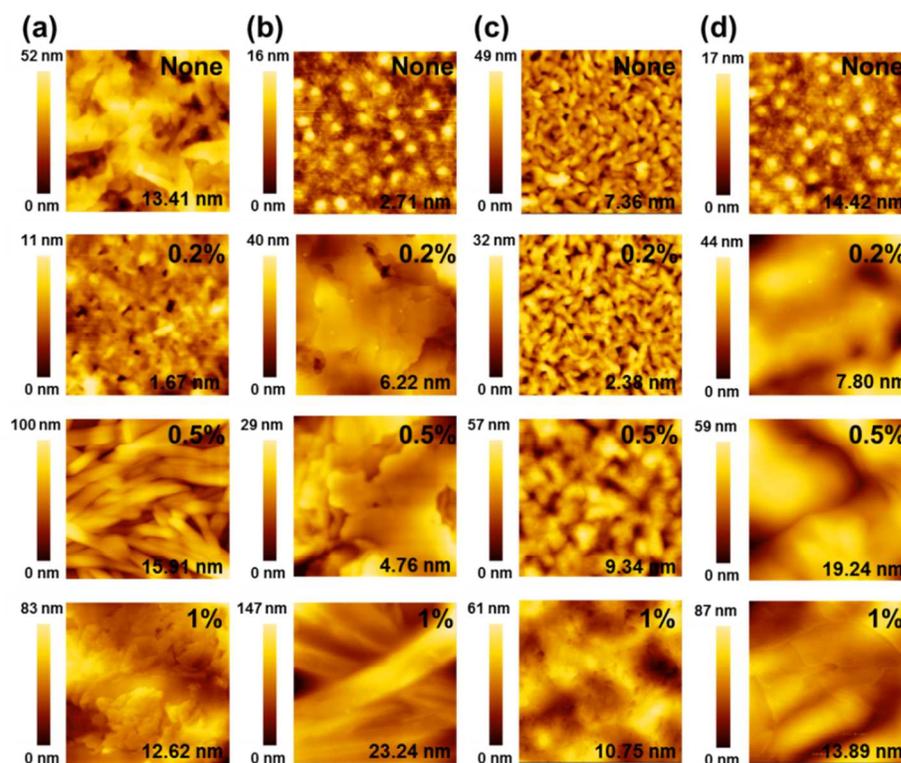


Fig. 3 AFM images (size : $2 \mu\text{m} \times 2 \mu\text{m}$) of (a) IDT, (b) ID2T, (c) ID3T and (d) IDED films as a function of DIO additive concentrations

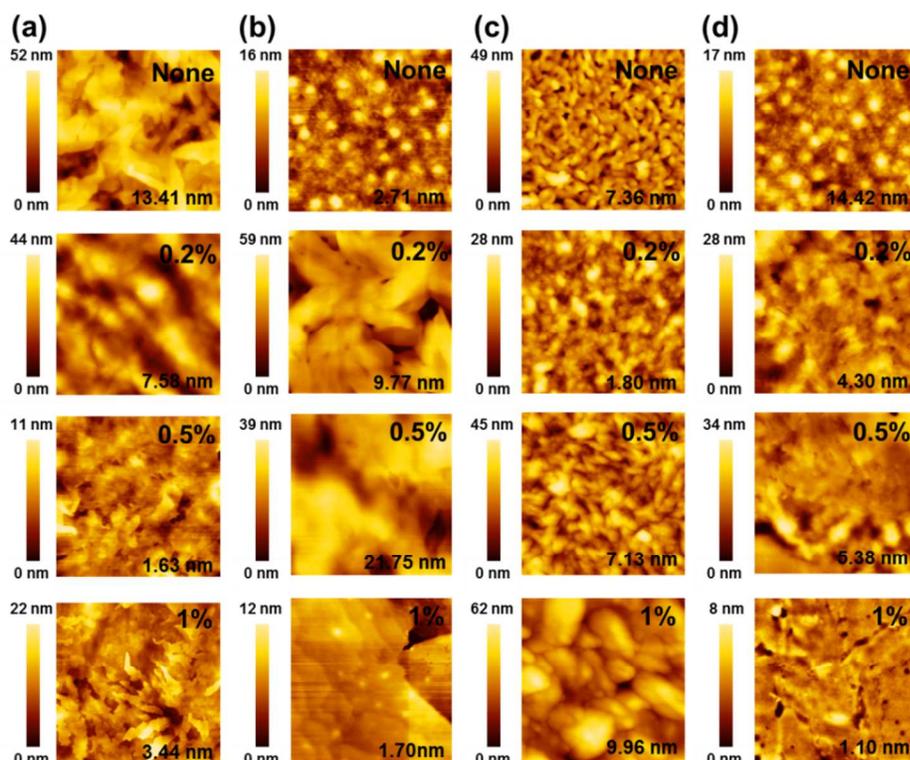


Fig. 4 AFM images (size : $2 \mu\text{m} \times 2 \mu\text{m}$) of (a) IDT, (b) ID2T, (c) ID3T and (d) IDED films as a function of CN additive concentrations.

3.28×10^{-4} to $1.37 \times 10^{-2} \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, when 0.2% CN was added. IDED FETs exhibited the lowest mobilities compared to the other isoindigo molecules at $9.77 \times 10^{-5} \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, while no benefit was observed by the use of solvent additives with this material. From AFM analysis, the addition of DIO and CN as a solvent additive markedly changed the morphologies of isoindigo films to form either more crystalline or smaller sized grains. The film morphologies correlated well to dramatic improvements in device performance. This study provides an understanding of the influence of volatile solvent additives and processing conditions on the morphology of small molecule systems, and will assist in the future selection of ideal processing additives in semiconducting devices utilizing small molecules.

Experimental

All OFETs were fabricated on heavily n-type doped Si substrate with a 200 nm SiO_2 gate insulator treated with OTS. The OTS solution was made by mixing OTS and toluene at a ratio of 1:100. SiO_2/Si substrates were soaked in the OTS solution for 10 min at 50°C hot plate in N_2 -glove box. The substrates were rinsed with toluene several times prior to use. 0.5 wt% isoindigo solutions were prepared in Chloroform without additive, with DIO (0.2%, 0.5%, 1%) and with CN (0.2%, 0.5%, 1%). The solutions were deposited by spin-coating at a spin rate of 2000 rpm for 60 s in N_2 -glove box. 70 nm-thick Au source and drain electrodes were deposited under vacuum by thermal evaporation on top of the semiconductor

layer using a shadow mask. The channel length (L) and width (W) were 40 and 200 μm , respectively. Electrical characterization was performed under a N_2 atmosphere using a Keithley SCS-4200 semiconductor parametric analyzer. More than 20 devices per each material were tested. AFM was carried out using a Seiko E-Sweep.

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Notes and references

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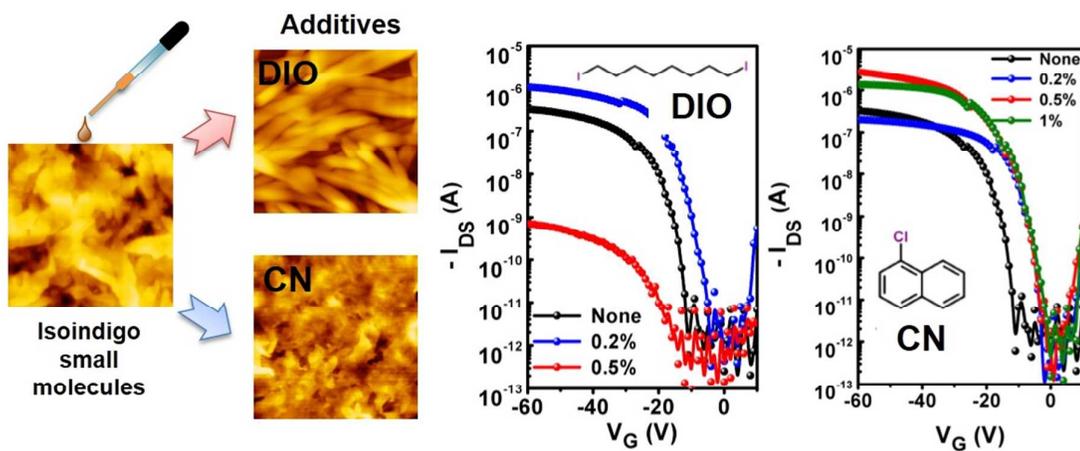
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†Electronic Supplementary Information (ESI) available: Output characteristics, UV-Vis absorption spectra, UPS spectra, DFT results, Energy levels, and XRD patterns. See DOI: 10.1039/b000000x/.

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We have investigated the field-effect transistor characteristics of a series of isoindigo based donor-acceptor small molecules via solvent additives.