INSTITUTE FOR POLYMER RESEARCH
**CELEBRATING 27 YEARS OF OFFICIAL INSTITUTE STATUS**
**THIRTY-THIRD ANNUAL SYMPOSIUM**
ON POLYMER SCIENCE/ENGINEERING
10 May, 2011



# High-Performance Semiconducting Polymer Materials for Printed Organic Electronics

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University of Waterloo

# Outline

- **Overview**
  - ▪Motivations for research on organic electronics
  - ▪Polymer semiconductors
- **Design of high-performance polymer semiconductors**
  - ▪Fused ring p-type polymers
  - ▪Donor-acceptor p-type polymers
  - ▪Ambipolar polymer semiconductors for CMOS-like logic circuits
- **Summary and future work**

# Why printed organic electronics?

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| | Silicon | Organic |
|---|---|---|
| Capital | $billions | $millions (Low investment) |
| FAB conditions | Ultra clean-room / High Temp. | Ambient / Low temp (Mild) |
| Process | Multi-step photolithography | Continuous direct printing (Simple) |
| Substrate | Rigid glass or metal | Flexible plastics (Robust) |
| Device size | << 1m$^2$ | 10 ft x roll to roll (Large area) |
| Cost | $100's / ft$^2$ | <$10 / ft$^2$ (Low cost) |


# Applications of printed organic electronics



RFID tags and smart labels

OLED lighting (GE)

Organic solar cells

E-paper (Plastic Logic)

Consumer electronics
(Nokia 888 concept cell phone)


Flexible displays (LG)

Market forecasts to 2027 - a $330 billion market (Source: IDTechEx)

# RESEARCH ACTIVITIES IN MY GROUP

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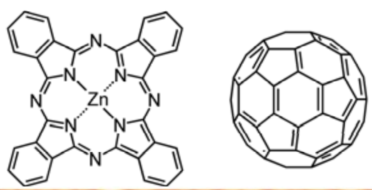
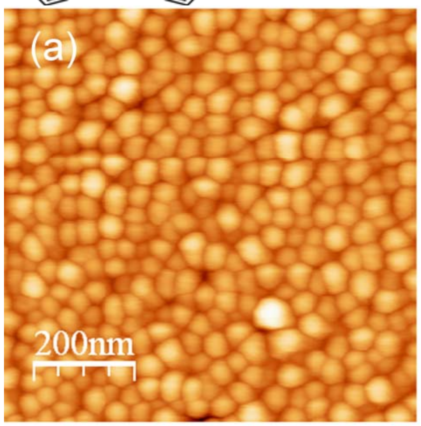
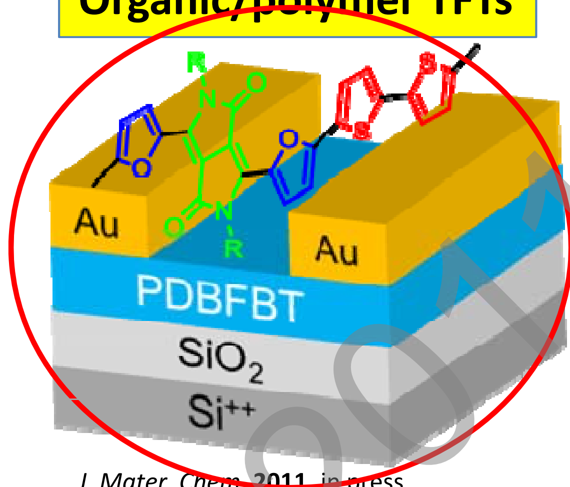
## Small molecule OPV



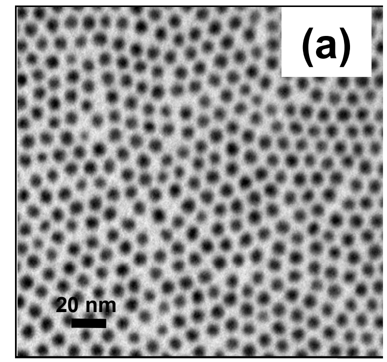

*Appl. Phys. Lett.* **2010**, *97*, 133304

## Organic/polymer TFTs



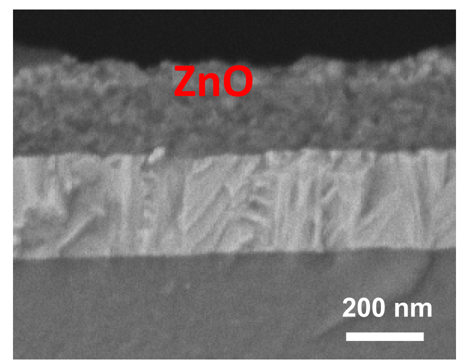

*J. Mater. Chem.* **2011**, in press.
*J. Am. Chem. Soc.* **2011**, *133*, 2198.
*Adv. Mater*. **2010**, *22*, 5409.
*Adv. Mater*. **2010**, *22*, 4862.

## Conductive inks



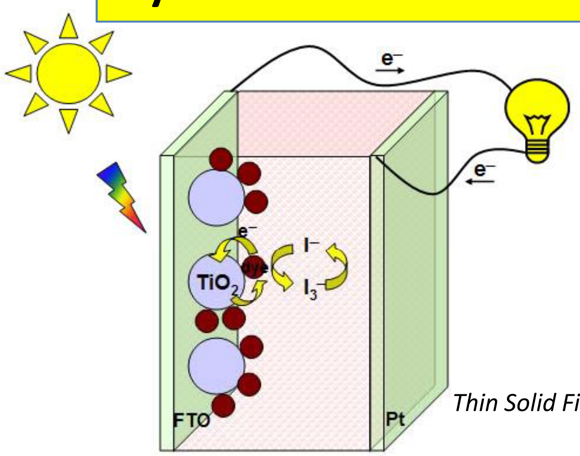

*J. Am. Chem. Soc.* **2007**, *129*, 1862.
*J. Am. Chem. Soc.* **2006**, *128*, 4202.
*J. Am. Chem. Soc.* **2005**,*127*, 3266.

## Printed inorganic TFTs



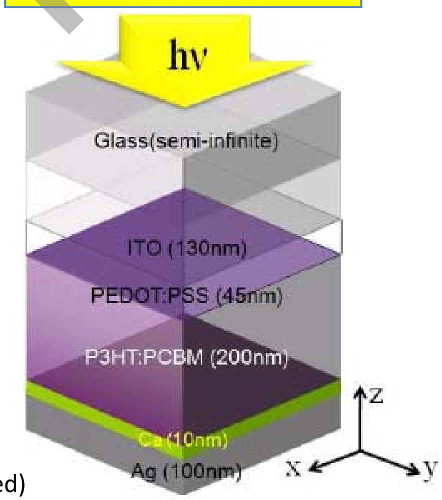

*J. Am. Chem. Soc.* **2007**, *129*, 2750.
*J. Appl. Phys*. **2007**, *102*, 076101.
*J. Phys. D: Appl. Phys.* **2008**, *41*, 125102.

## Dye-sensitized solar cells




*Thin Solid Films* (**2011**, accepted)

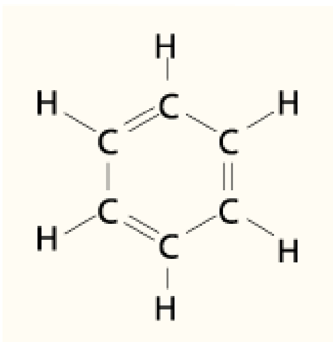
## Polymer BHJ PV




*Energy Env. Sci.* **2011**, in press.
*Sol. Ener. Mat. Sol. Cells* **2010**, 94, 1618.

# π-Conjugated polymers

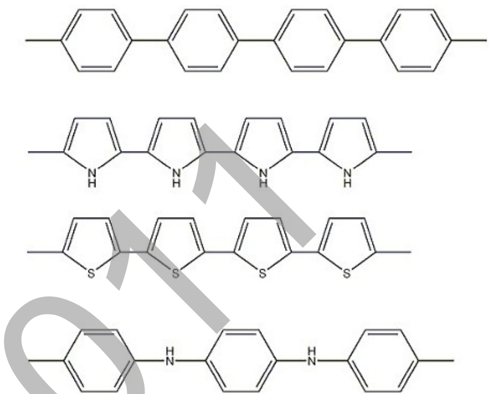
## π-electron delocalization in benzene

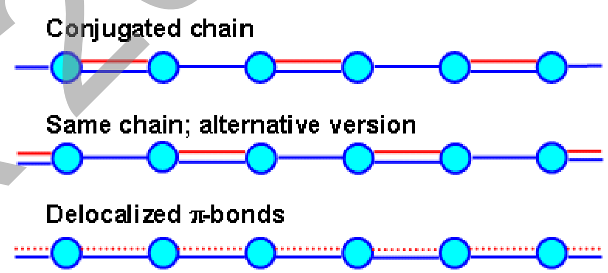


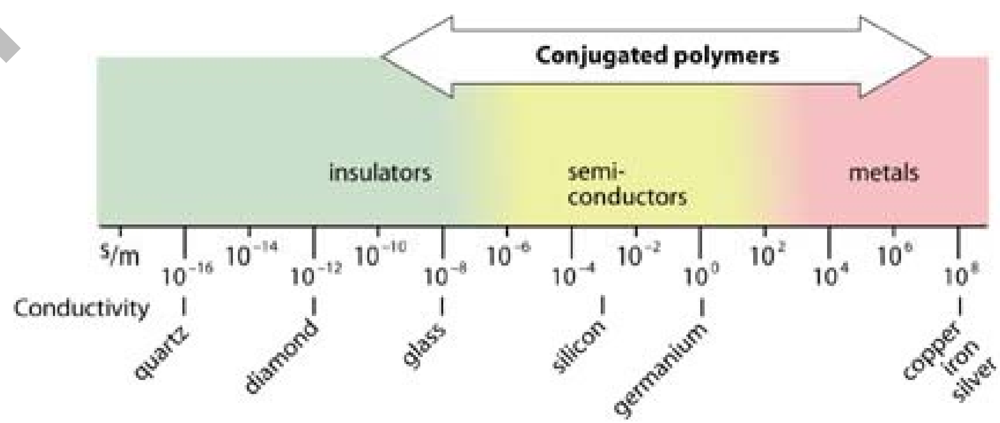


Molecular orbital shaped like a doughnut, above and below the ring, extending over all six carbon atoms.

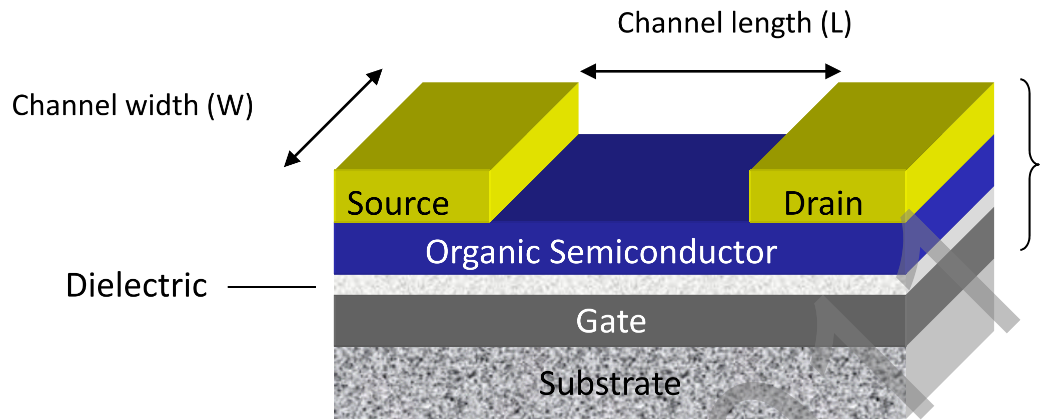
## π-conjugated polymers

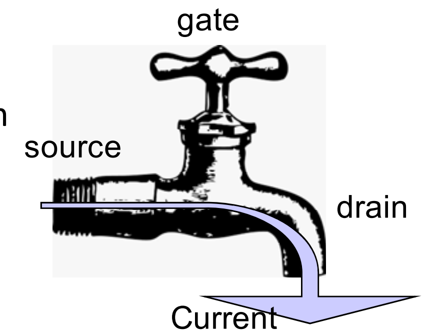


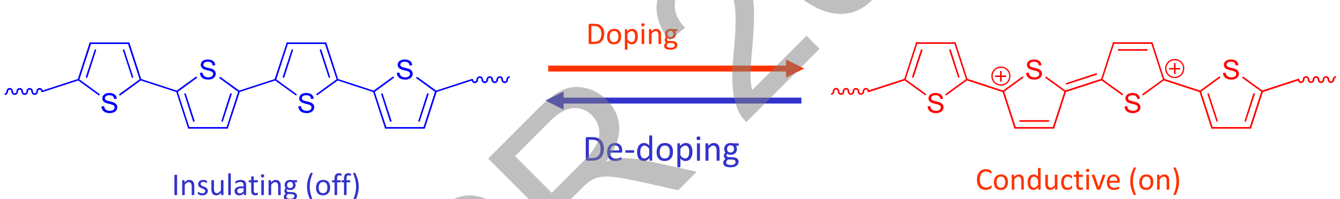


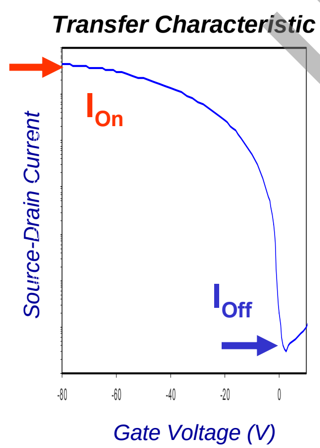



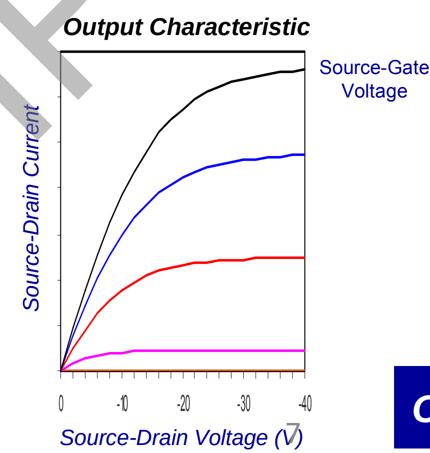
# Organic thin film transistors (OTFT)




***Field-effect Mobility ($\mu$)***

***Linear Regime*** ***($V_d < V_g$):***

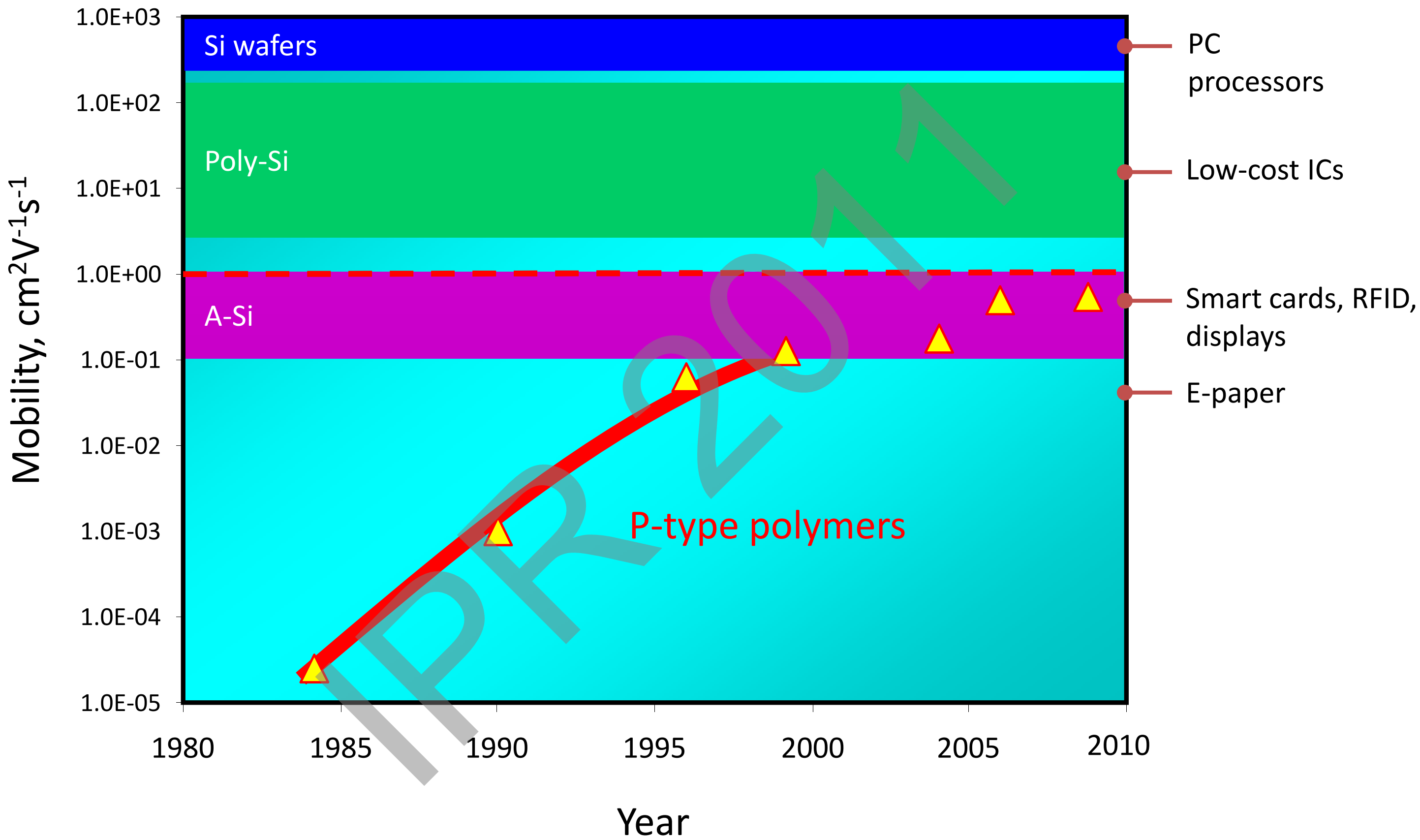
$I_d = V_d C_i \mu (V_g - V_T) W/L$

***Saturation Regime*****: ($V_d > V_g$):**

$I_d = C_i \mu (V_g - V_T)^2 W/2L$

***Current on/off ratio ($I_{on} / I_{off}$)***

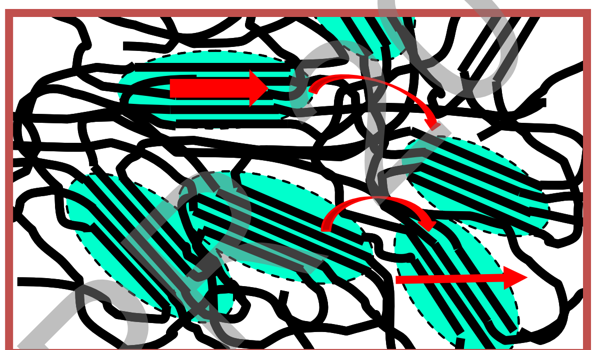
# Mobility of p-type polymer semiconductors




Is 1 $cm^2/V.s$ an upper limit?

# Charge Carrier Transport in Polymer Semiconductors

- Polymer semiconductors always comprise disordered amorphous regions
- Weak ver der Waals bonds between polymer chains:
  - Large intermolecular distance (π: ~3-4 Å)
  - Charge transport through hopping of charges between localized states
  - Upper limit: ~1 $cm^2/V.s$ (G. Horowitz, *Adv. Mater.* 1998, *10*, 365)



**Three modes of charge carrier transport at different levels**

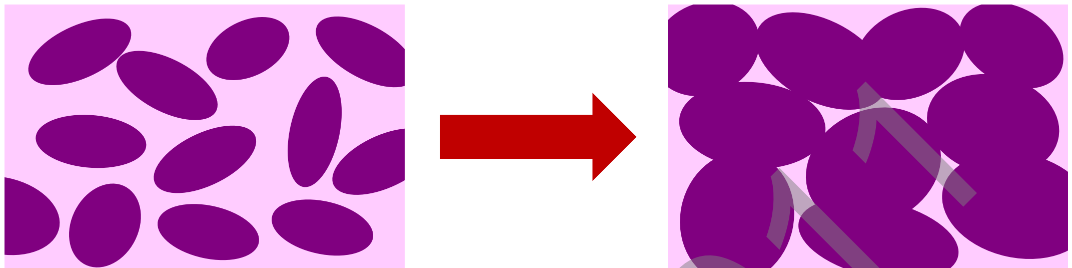
**Intramolecular**: Can be very fast (~$10^3$ $cm^2/V.s$); determined by coplanarity

**Intermolecular (interchain)**: Slow (up to ~10 $cm^2/V.s$); determined by overlapping area and distance

**Intergranular (interdomian)**: Very slow (can be ~$10^{-5}$ $cm^2/V.s$ or lower); determined by crystallinity/morphology

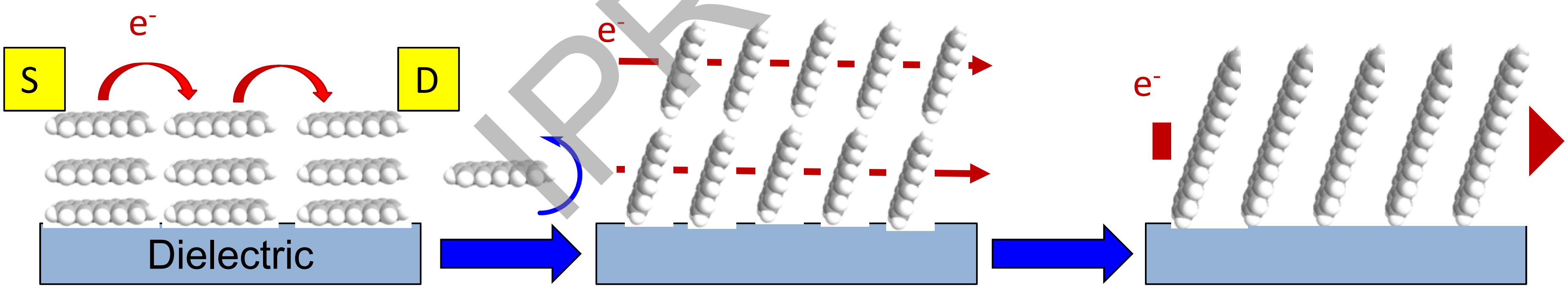
# Molecular organization

1. Crystallinity and crystal size



- Higher crystallinity and larger crystal domains yield high mobility

2. Molecular packing motif



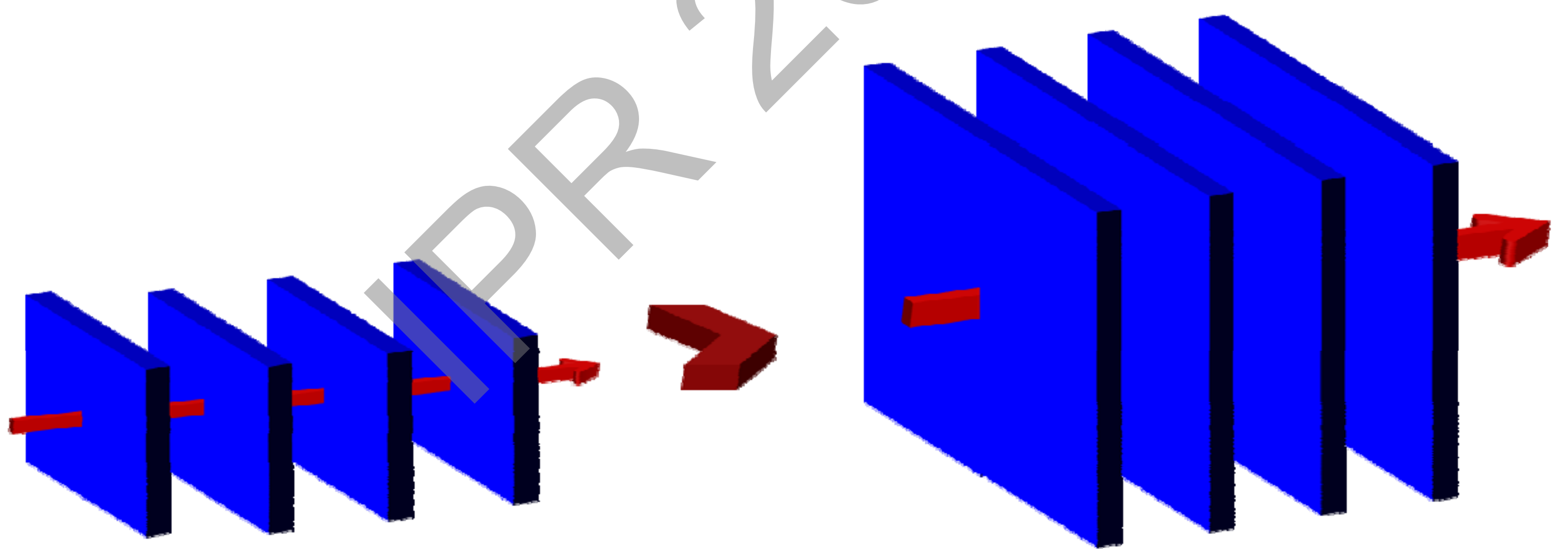

Edge-down (face-on) | **Edge-on** | Larger π-overlap

- Smaller π-π distance, edge-on orientation, and larger π-π overlap lead to higher mobility

# Improving *Intermolecular* and *Intergranular* charge carrier transports

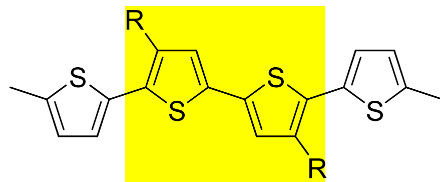
Strategy 1: Use of fused aromatic rings

- Stronger π-π stacking force
- Large π-π overlap
- Increased crystallinity



# Thiophene-based polymers with fused ring structures

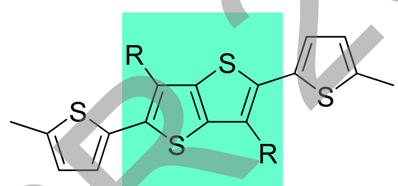
Thiophene




PQT

Ong, B. S., et al, *J. Am. Chem. Soc.* 2004, *126*, 3378.

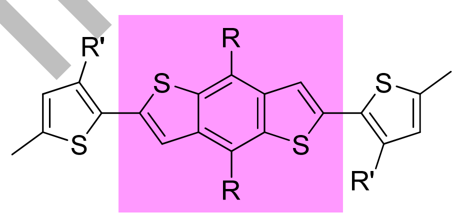
Thieno[3,2-b]thiophene
(TT)




PBTT

Li, Y., *et al*, *Adv. Mater.* 2006, *18*, 3029.

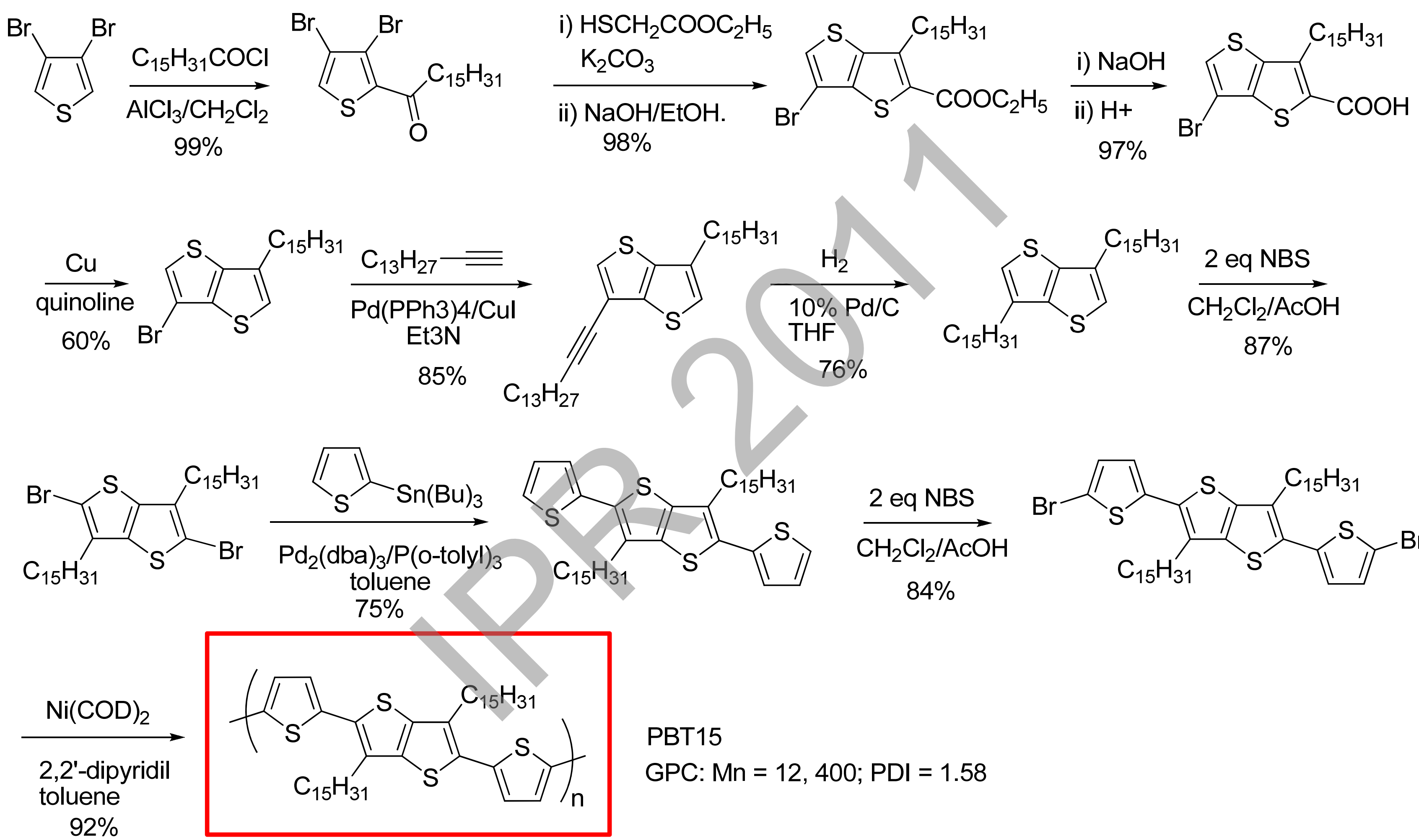
Benzo[1,2-b:4,5-b']dithiophene
(BDT)




PBBDT

Pan, H., *et al*, *J. Am. Chem. Soc.* 2007, *129*, 4112.

# Synthesis of PBTT




PBT15
GPC: Mn = 12, 400; PDI = 1.58

10 steps; total yield: 18%

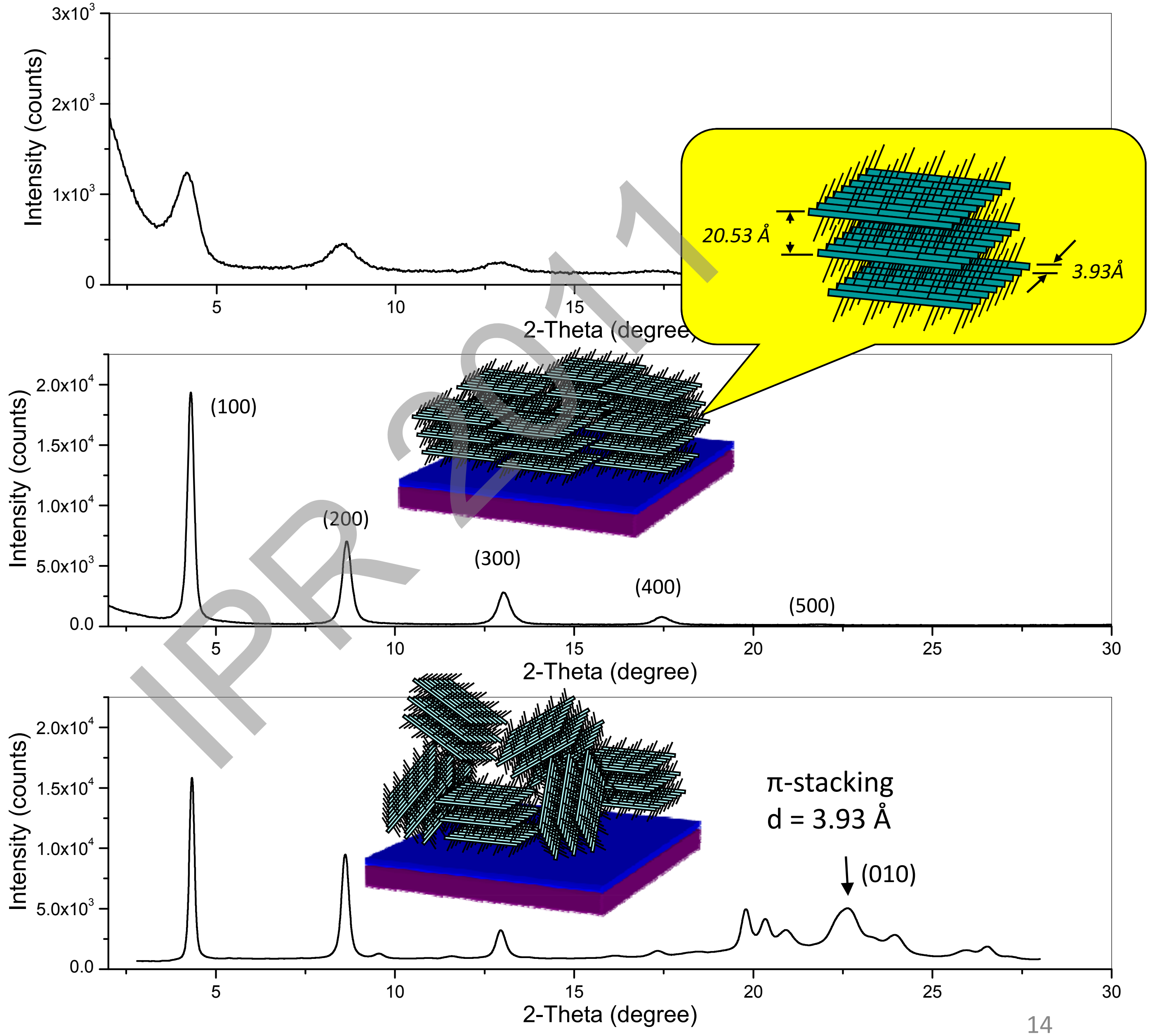
Li, Y.; Wu, Y.; Liu, P.; Birau, M.; Pan, H.; Ong, B. S. *Adv. Mater.* 2006, *18*, 3029.

# Molecular ordering of PBTT revealed by XRD

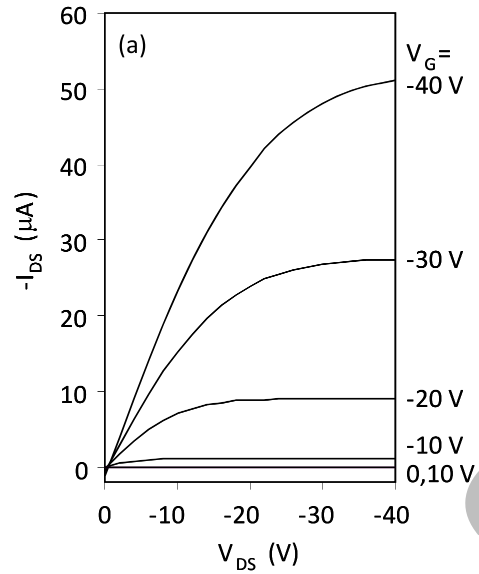
As-cast film

Annealed film
(150 ° C/10min)

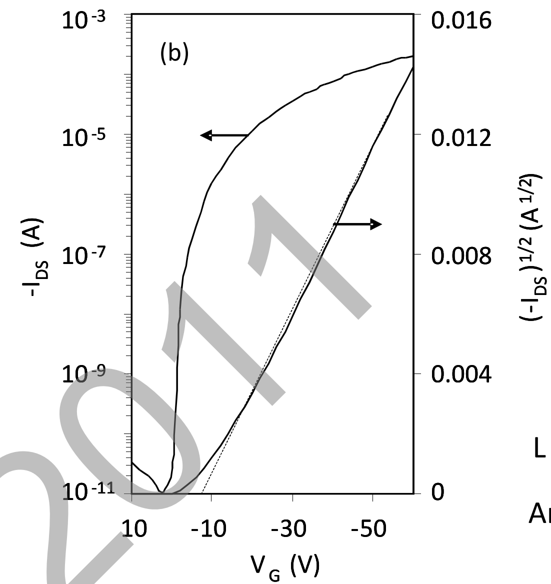
Annealed powder
(150 ° C/10min)



# OTFT performance of PBTT




Output curves




Transfer characteristics ($V_{ds}$ = -60 V)

L = 90 μm; W = 5000 μm

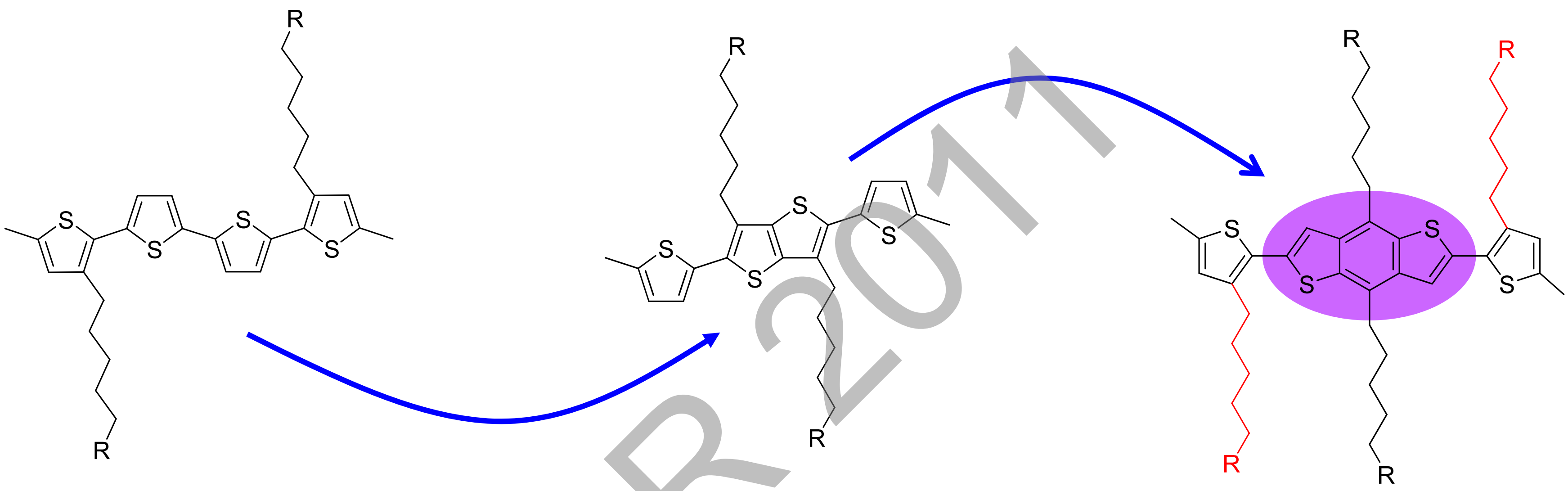
Annealed at 150 ° C

| | Mobility, $cm^2/V.s$ (μ) | Current on/off ratio ($I_{on}/I_{off}$) |
|---|---|---|
| Without annealing | ~0.10 | $10^6$ |
| Annealed at 150 ° C | ~0.25 | $10^7$ |

Li, Y.; Wu, Y.; Liu, P.; Birau, M.; Pan, H.; Ong, B. S. *Adv. Mater.* 2006, *18*, 3029.

- High crystallinity, favored molecular organization and large π-overlap are contributable to the high mobility
- Similar mobility to that of PQT and P3HT even without thermal annealing

# Benzo[1,2-b:4,5-b’]dithiophene (BDT) building block



PQT

$\mu_h$= ~0.12 cm²/V.s

PBT

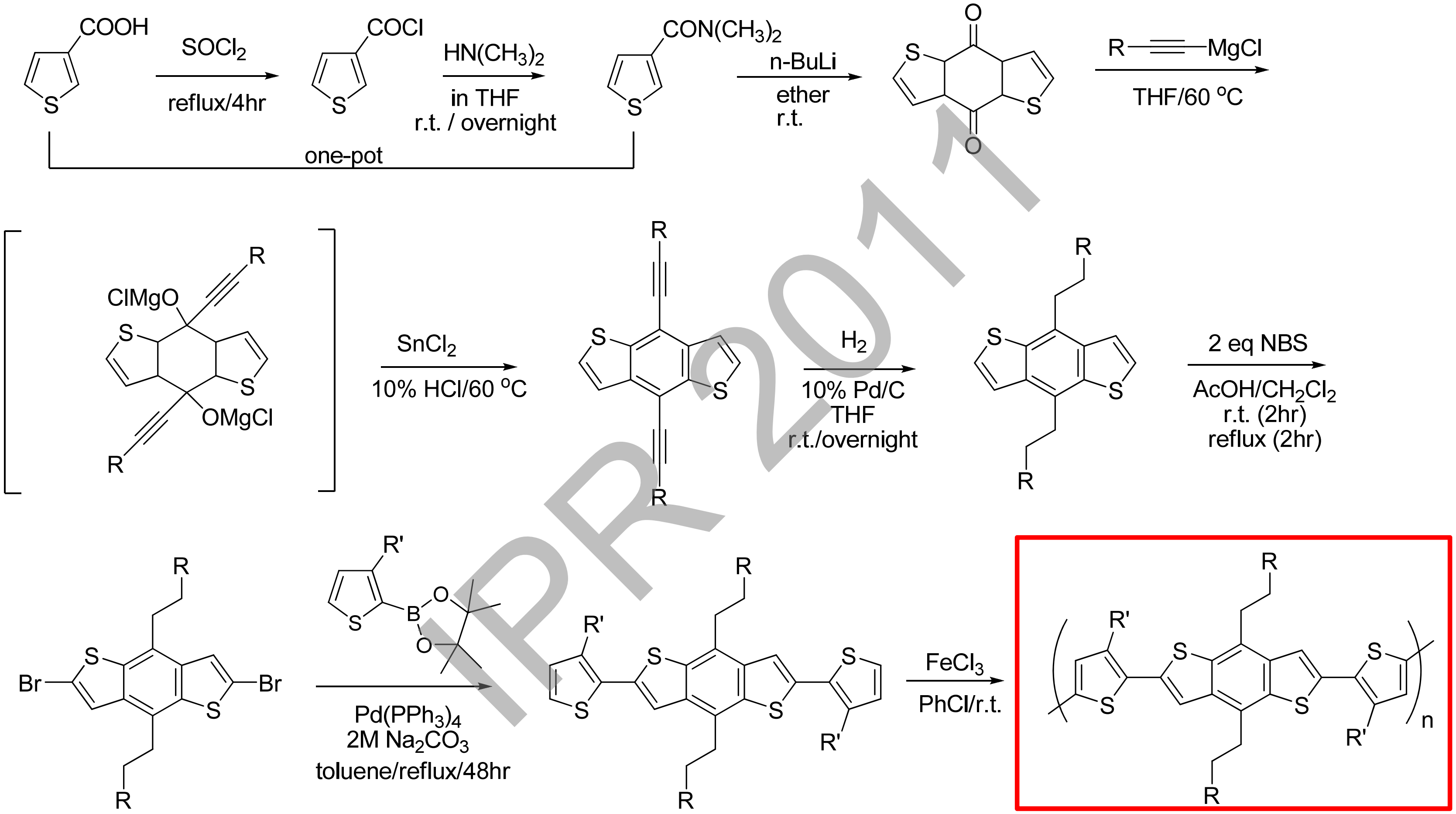
$\mu_h$= ~0.12 cm²/V.s

PBBDT

$\mu_h$= ?

- More extended fused ring (larger π-overlap)
- Stronger backbone interaction
- Additional side chains for better solubility

# Synthesis of PBBDT

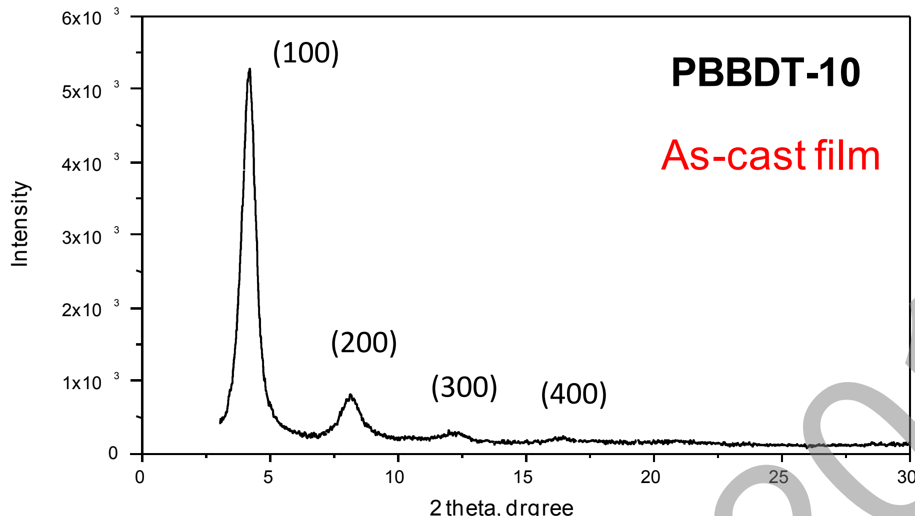



PBBDT-6 (R=C4;R'=C6)
PBBDT-10 (R=C8;R'=C10)
PBBDT-12 (R=C10; R'=C12)
GPC: $M_n$ = 16 -274 kD

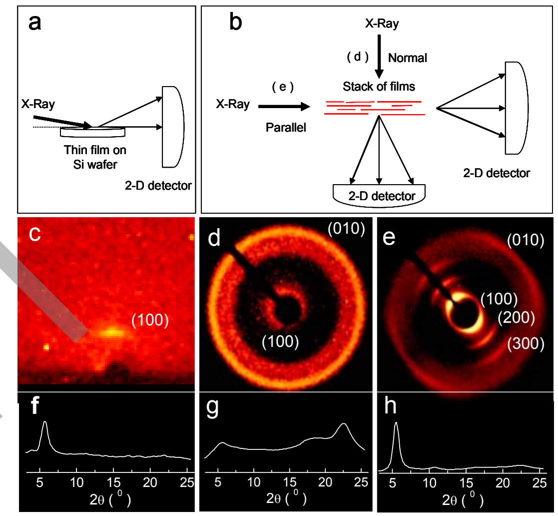
9 steps; total yield: 11%

Pan, H.; Li, Y.; Wu, Y.; Liu, P.; Ong, B. S.; Zhu, S.; Xu, G. *J. Am. Chem. Soc.* 2007, *129,* 4112.

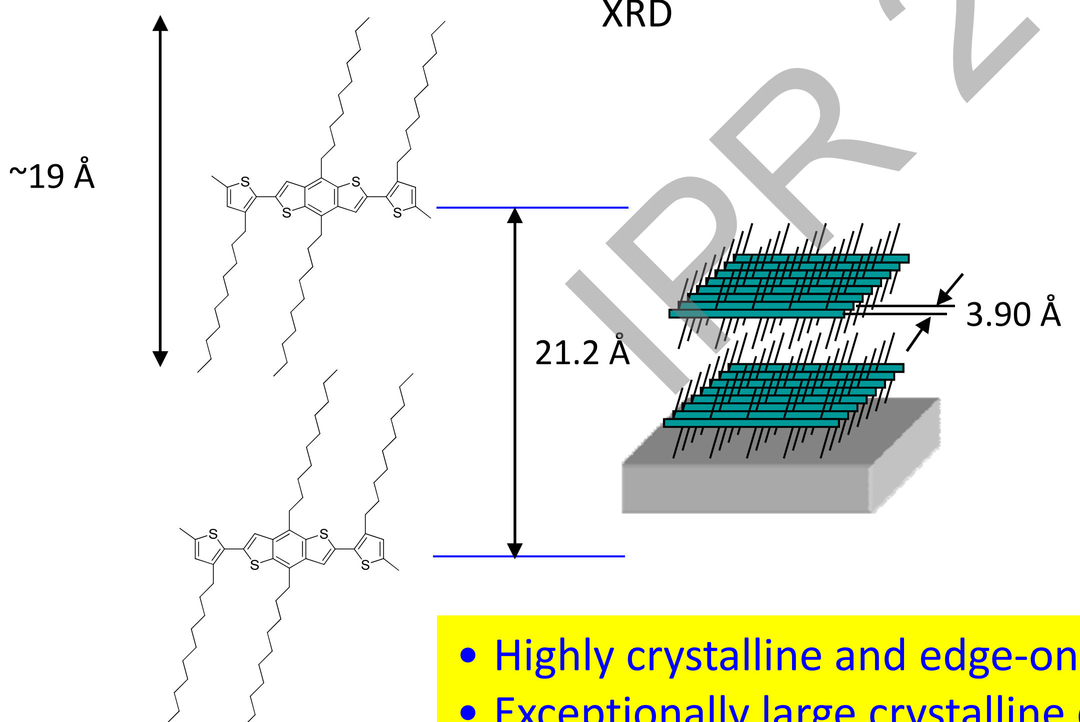
# Molecular organization

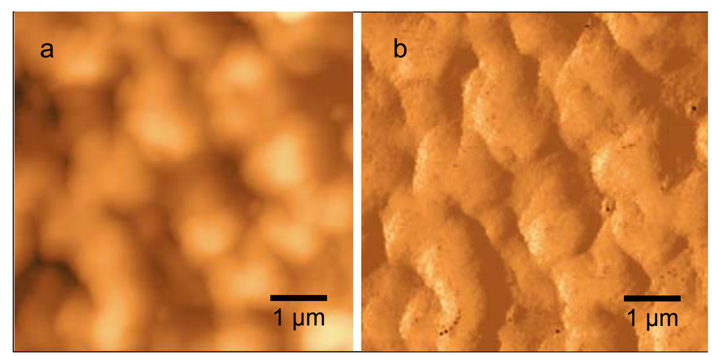


XRD



2-D XRD



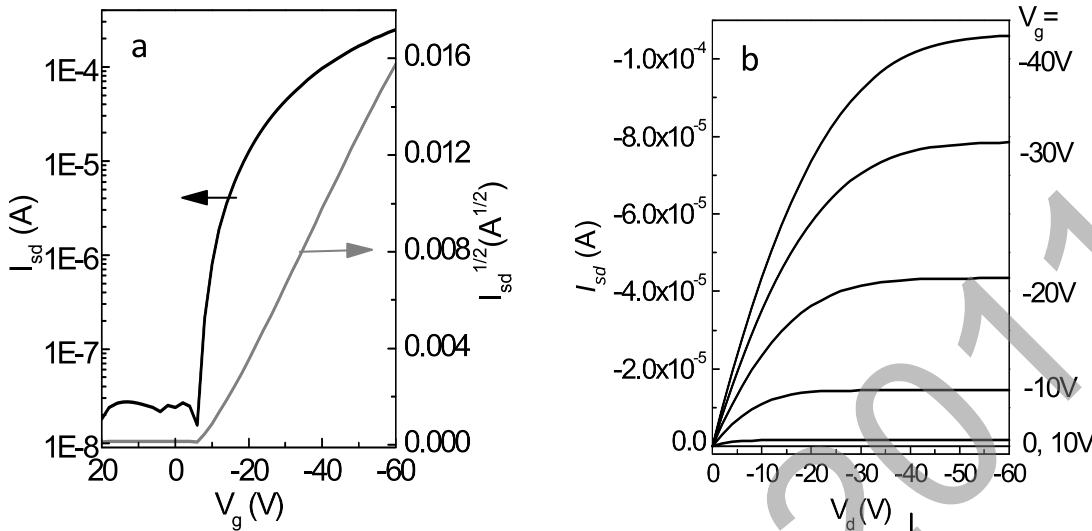



AFM

Large crystal domains (~μm's)

- Highly crystalline and edge-on orientation w/o annealing
- Exceptionally large crystalline domains

# TFT performance



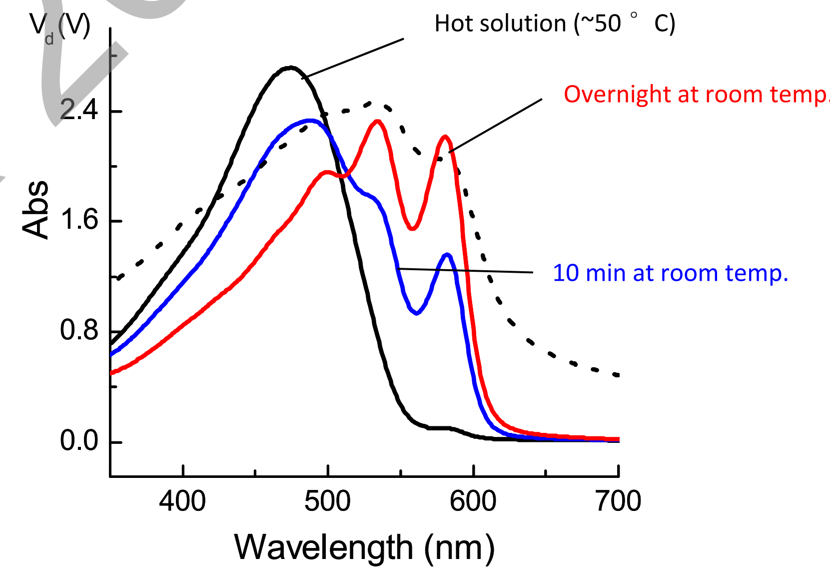

I-V characteristics of an illustrative as-prepared TFT device using PBBDT semiconductor (channel length = 90 μm; channel width = 1000 μm): (a)transfer curve in saturated regime at a constant source-drain voltage of -60 V and square root of absolute value of drain current as a function of gate voltage; and (b)output curves at different gate voltages.

Without annealing:

- $\mu$ = ~0.40 $cm^2V^{-1}s^{-1}$
- $I_{on}/I_{off} = 10^5$

Annealing (up to 200 °C):

- No improvements




UV-vis spectra of PBBDT.

- Poor solubility at room temperature
- Must be processed using hot solutions

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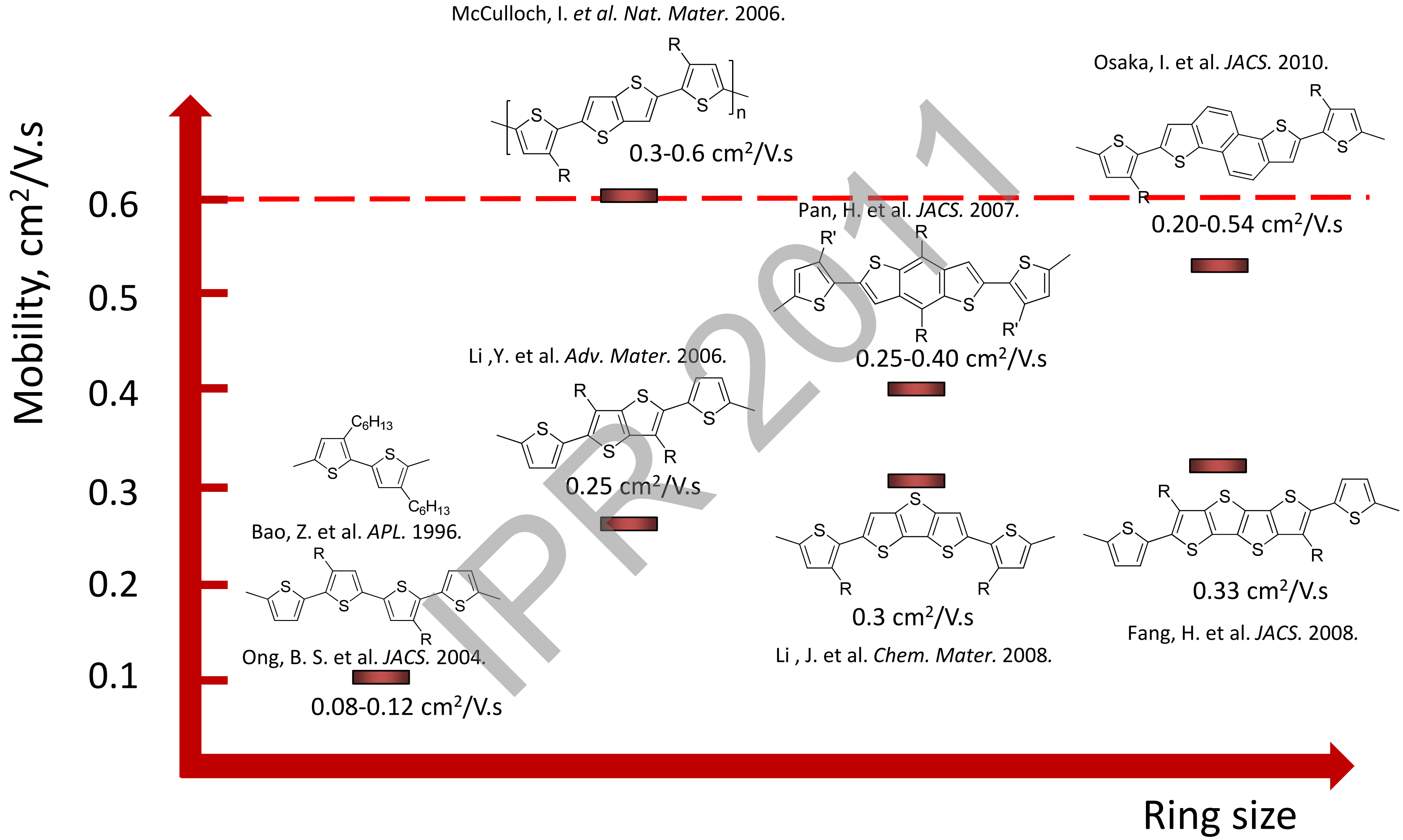
# Exemplary Fused-ring-containing polymers

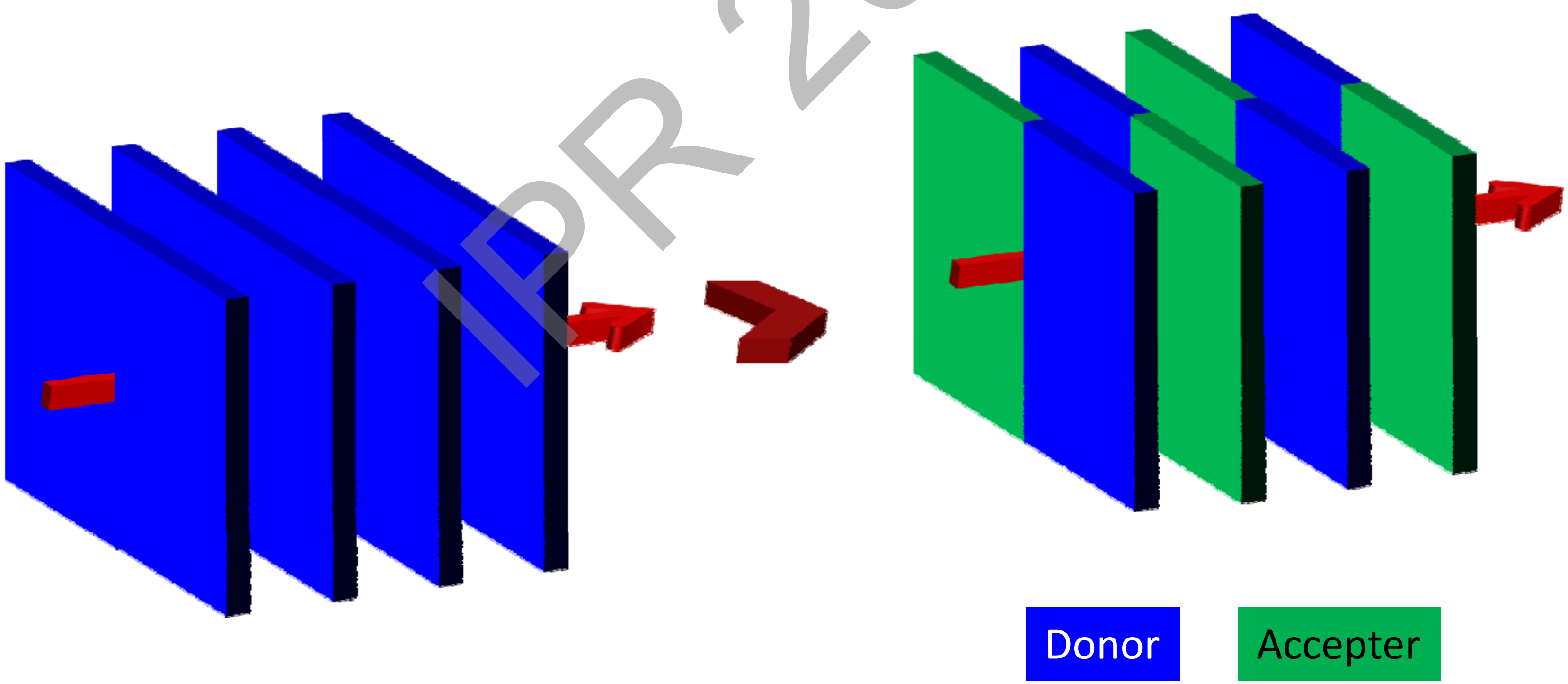




- Improvements in mobility by using fused rings are limited.
- Solubility decreases as ring size increases

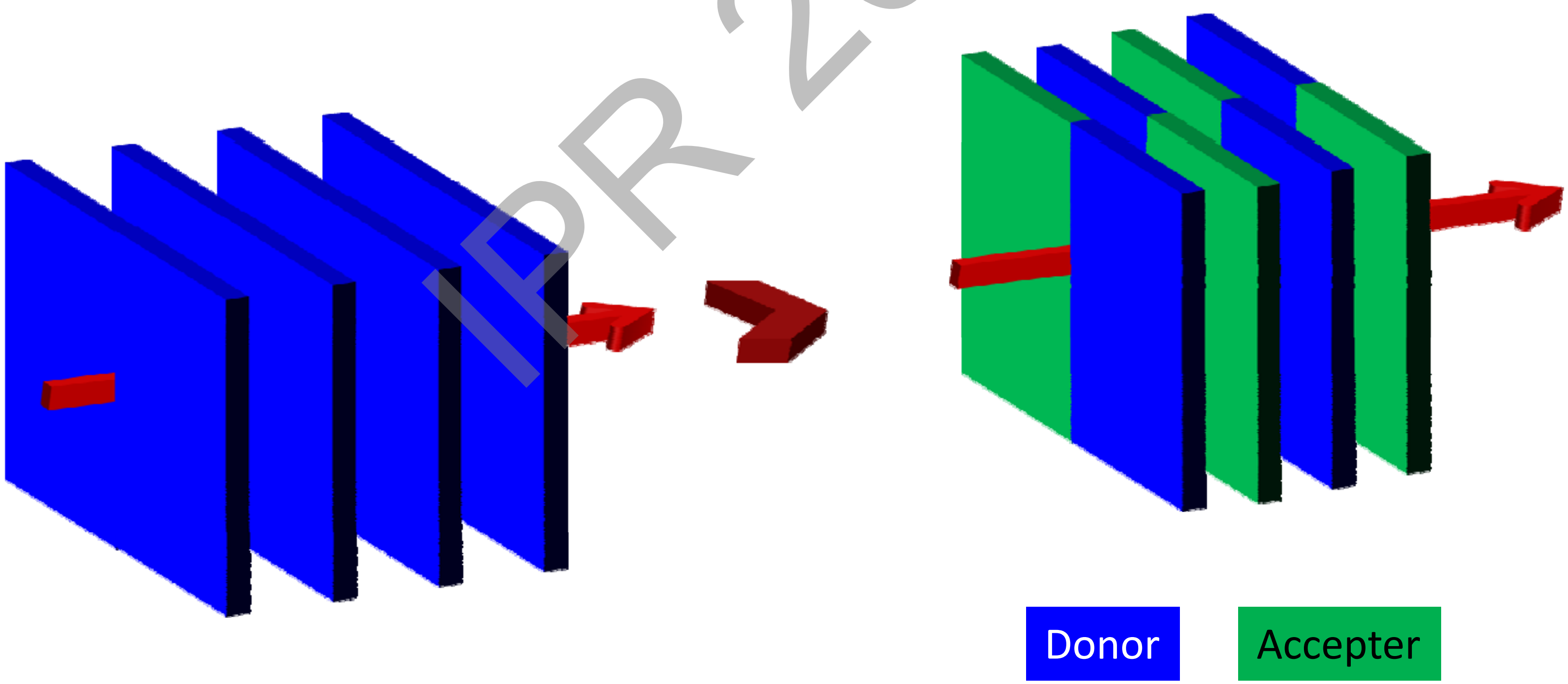
# Improving *Intermolecular* and *Intergranular* charge carrier transports

Strategy 2: Intermolecular donor-acceptor interactions



# Improving *Intermolecular* and *Intergranular* charge carrier transports

Strategy 2: Intermolecular donor-acceptor interactions



# Diketopyrrolopyrrole (DPP)-based D-A polymers

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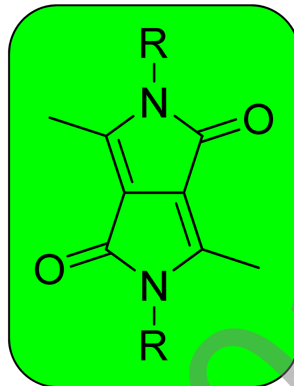
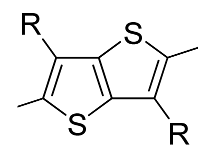
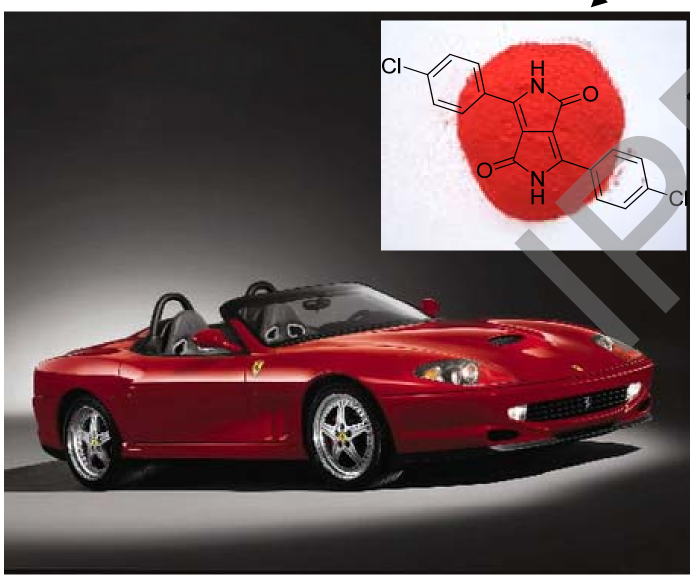
## -*Fused ring* + *D-A interactions*



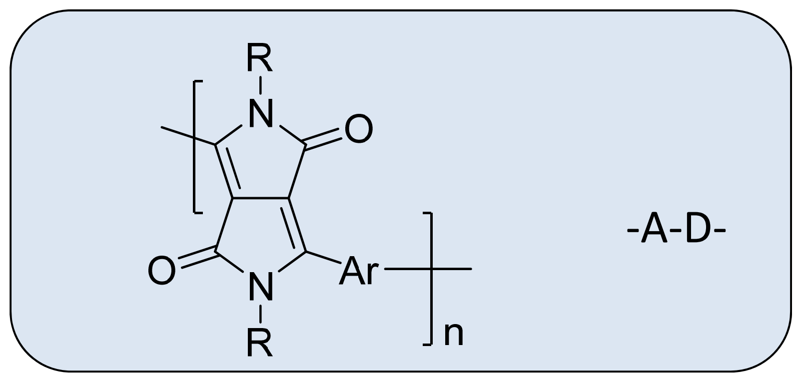

- An electron-accepting moiety
- Fused ring analog to TT




- Very stable in air

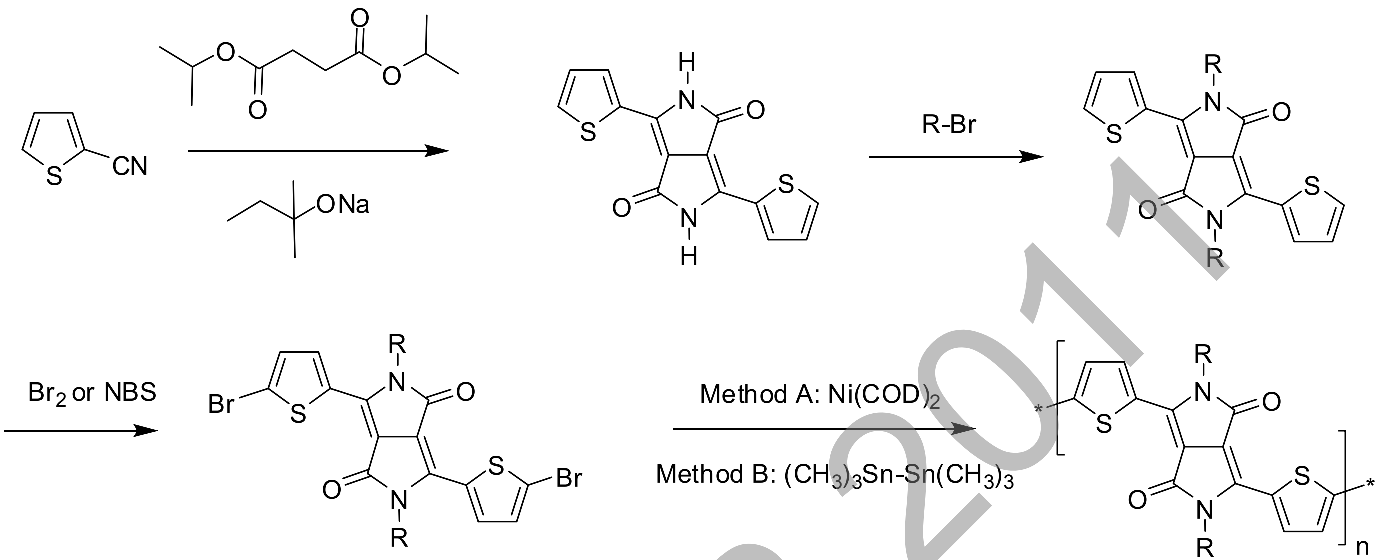


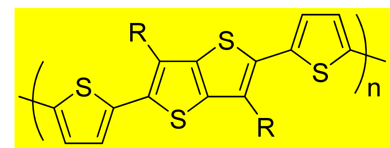

Pigment red 254 or 'Ferrari Red'




Y. Li, U. S. Patent Application 2009/65766 A1 (Xerox)
Y. Li, U. S. Patent Application 2009/65878 A1 (Xerox),
Y. LI, U. S. Patent 7910684 (Xerox)
Y. Li, U. S. Patent 7932344 (Xerox)

# PBTDPP



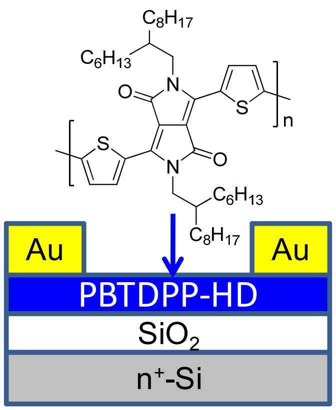

PBTT

| R | Polymn method | Solubility | Yield, % | $M_w/M_n$ |
|---|---|---|---|---|
| 1-Dodecyl (C12) | A,B | Insoluble | Trace | - |
| 1-Hexadecyl (C16) | A | Very poor | Trace | - |
| | B | Hot CB | 62% | 2590 (HT-GPC) |
| 1-Octadecyl (C18) | A | Insoluble | Trace | - |
| | B | Hot CB | 63% | 2820 (HT-GPC) |
| 2-Hexyldecyl (C16) | A | Good | 24% | 67,070/28,100 |
| 2-Octyldodecyl (C20) | A | Very good | 99% | 392,770/146,290 |

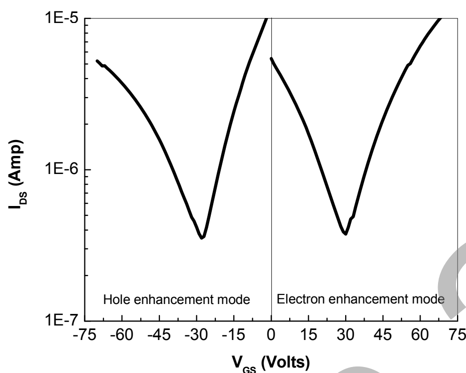
Y. Li, et al., unpublished results (2006-2009)

# OTFT Performance

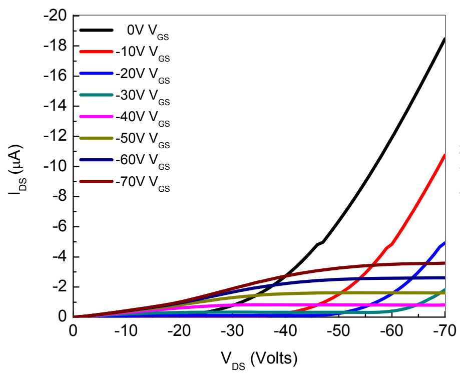
- Typical ambipolar transport characteristics



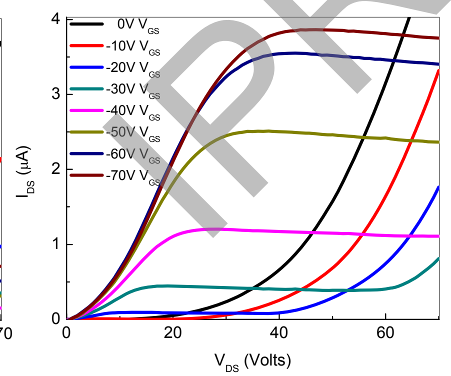

(a)




(b)



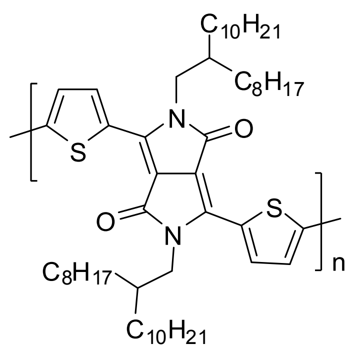

(c)



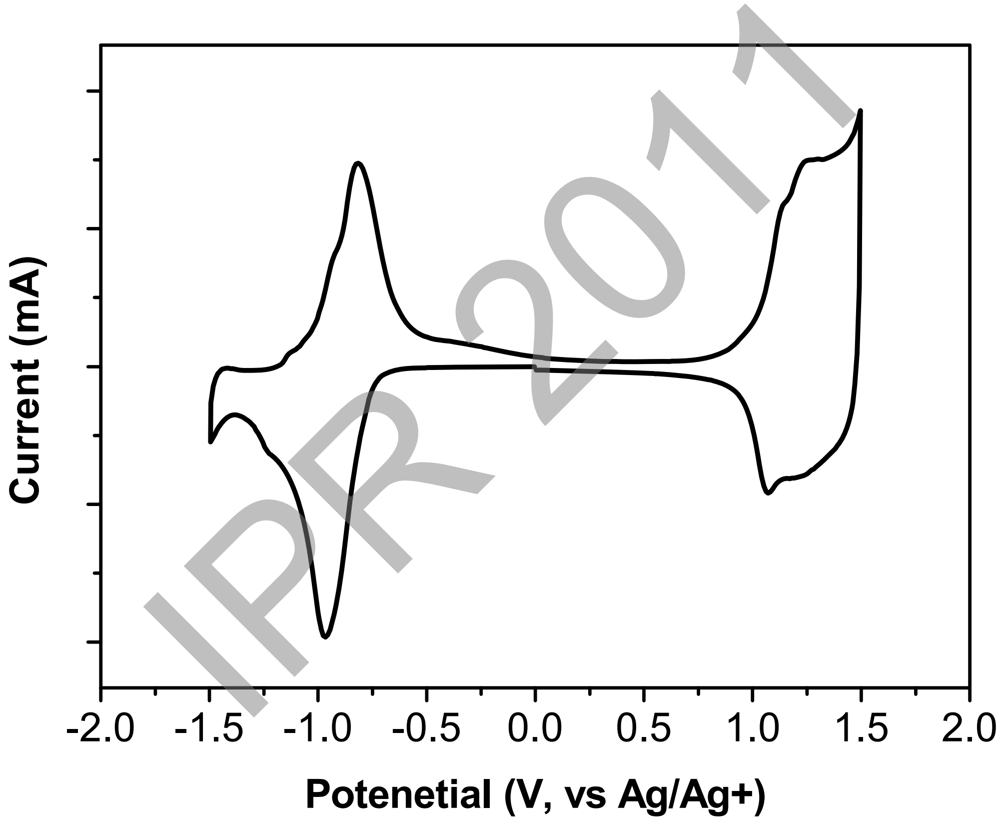

(d)

| Polymer | Annealing temperature, ◦C | Hole mobility, $cm^2/V.s$ | Electron mobility, $cm^2/V.s$ |
|---|---|---|---|
| PBTDPP-HD | **r. t.** | **0.024** | **0.056** |
| | 100 | 0.017 | 0.057 |
| | 140 | 0.013 | 0.036 |
| PBTDPP-OD | r. t. | 0.006 | 0.025 |
| | 100 | 0.013 | 0.010 |
| | 140 | 0.013 | 0.007 |

# Cyclic Voltammetry



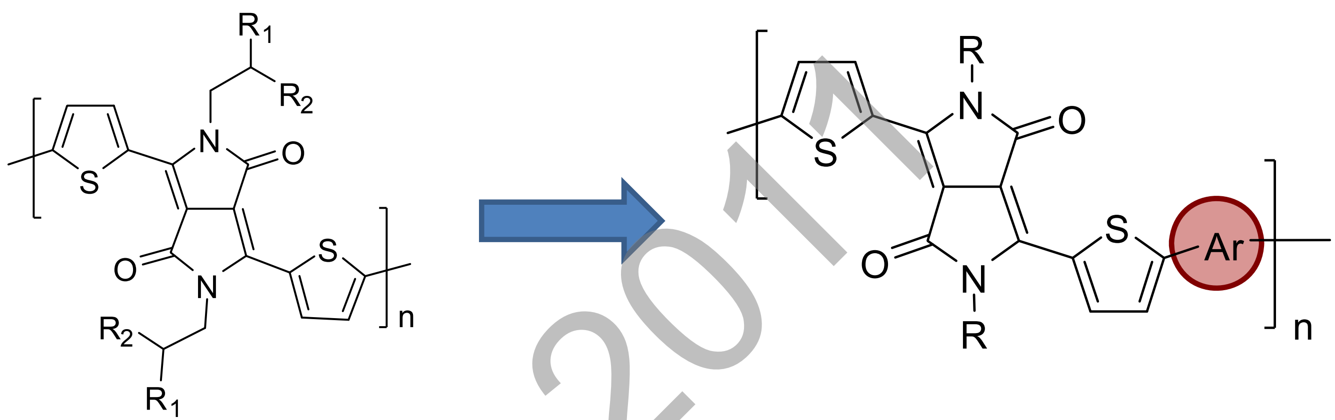

**PBTDPP-OD**



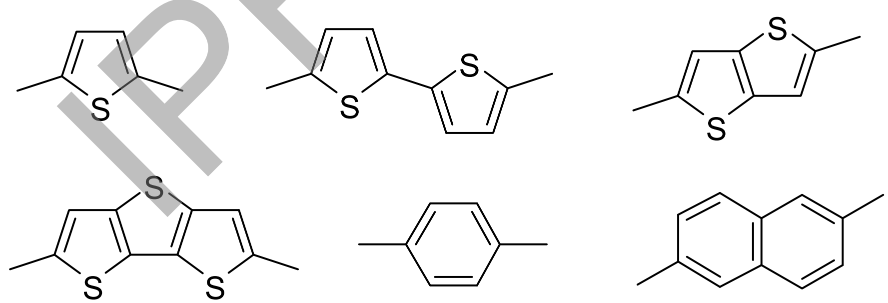


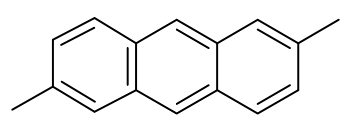

- Reversible oxidative and reductive processes
- Might be good for both hole and electron transports

# P-Type DPP-based Polymers



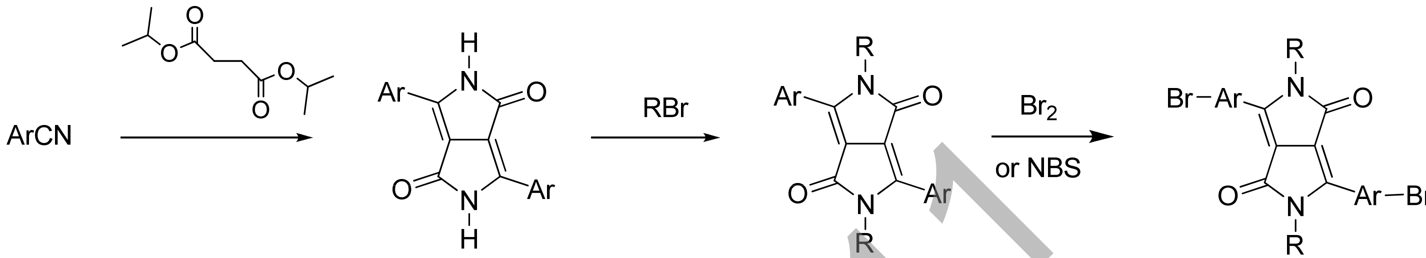

Ar: An electron donor

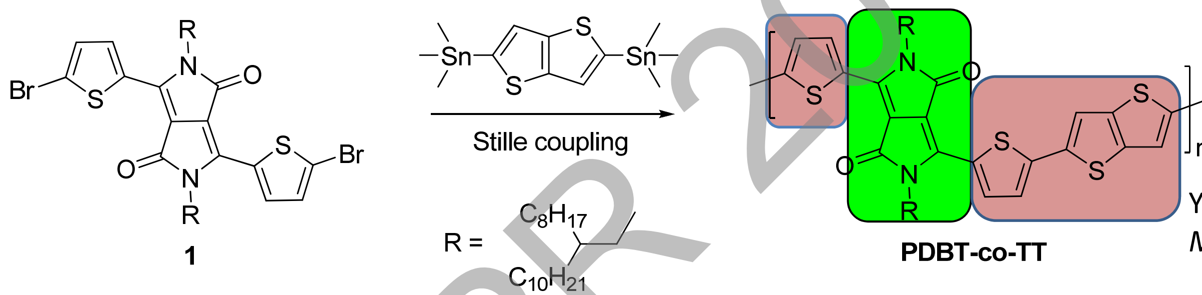


etc.

# PDBT-co-TT polymer



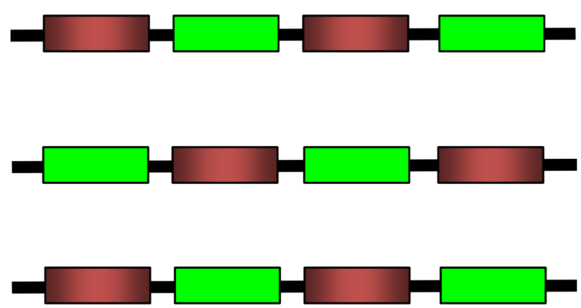

Yield = 95.4%

$M_w/M_n$ (GPC) = 211,300/89,700

Y. Li, S. P. Singh, P. Sonar, *Adv. Mater*. 2010, 2010, *22*, 4862.

Elctron donor

Elctron acceptor



Strong interchain interactions

- π-π distance is expected to be reduced due to DA interactions

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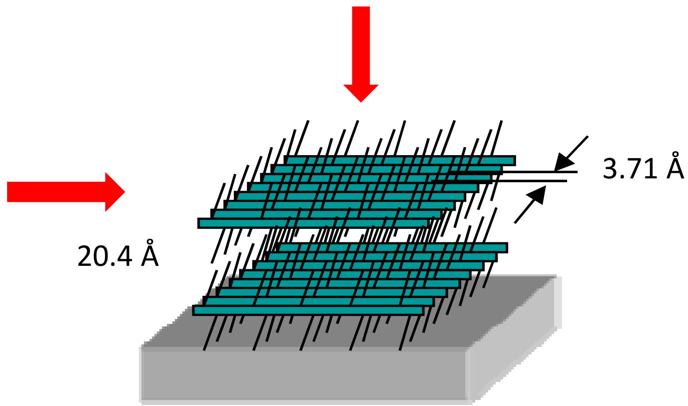
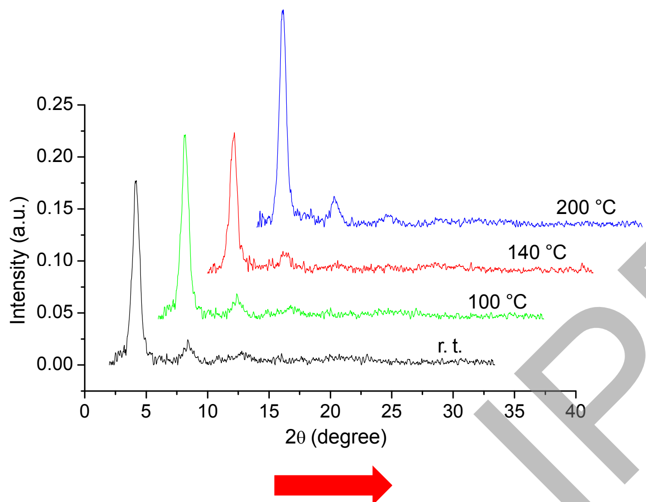
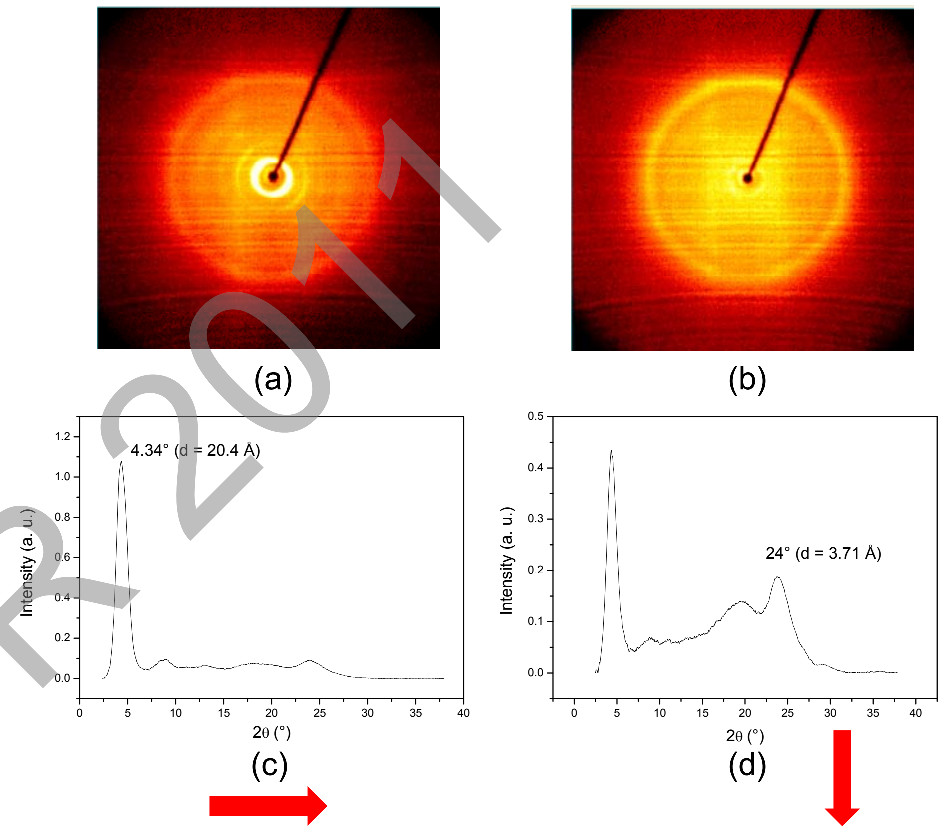
# Molecular organization of PDBT-co-TT




XRD of spin-coated thin films

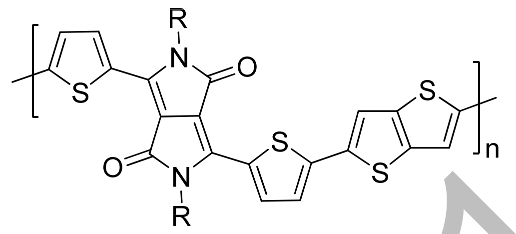



2-D XRD of a stack of thin films

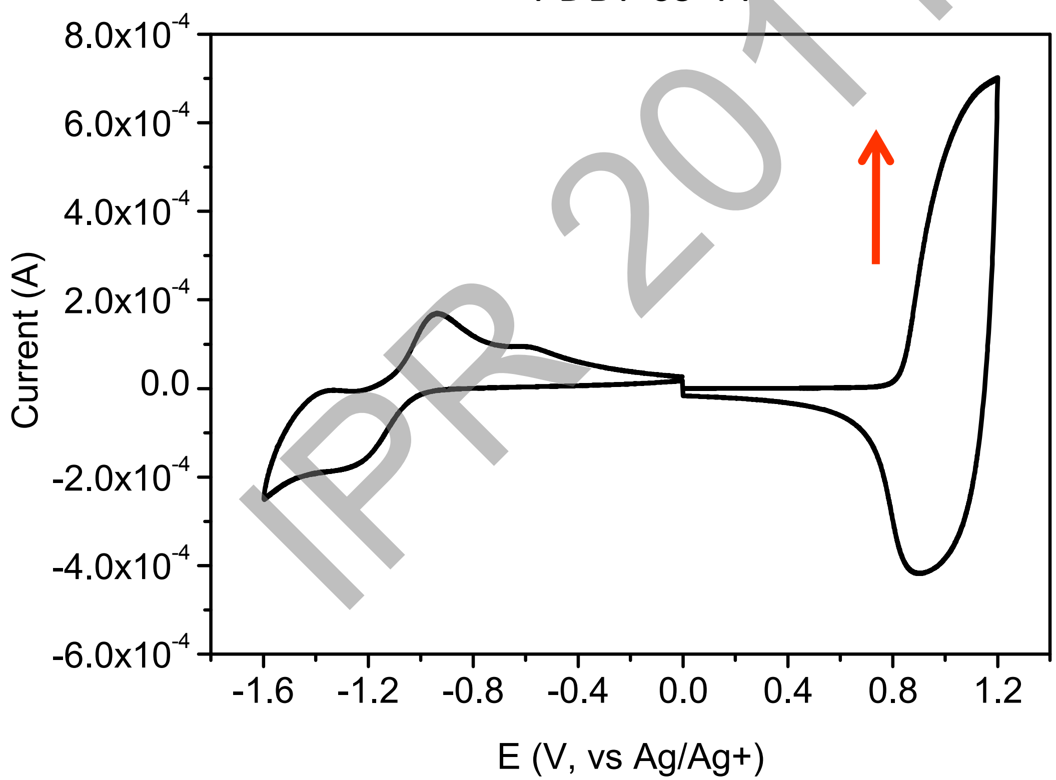


- Small π-π distance of 3.71 Å: Good for interchain charge transport
- High crystallinity of as-cast films: Strong self-assembly ability
- Crystallinity is not very sensitive to annealing temperature

# Cyclic Voltammetry



PDBT-co-TT



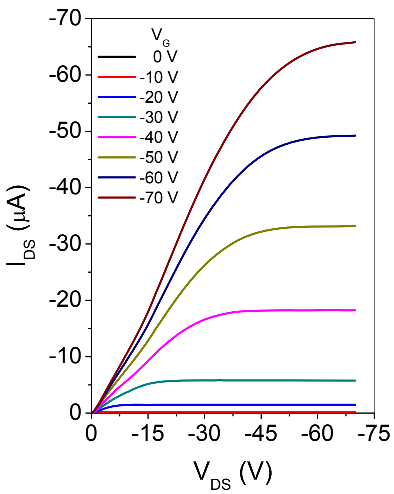


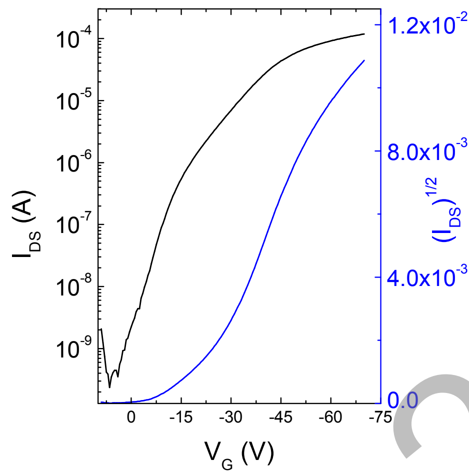

HOMO: 5.25 eV

LUMO: 3.40 eV (4.02 eV from UV-vis)

# OTFT performance of PDBT-co-TT

## Without annealing



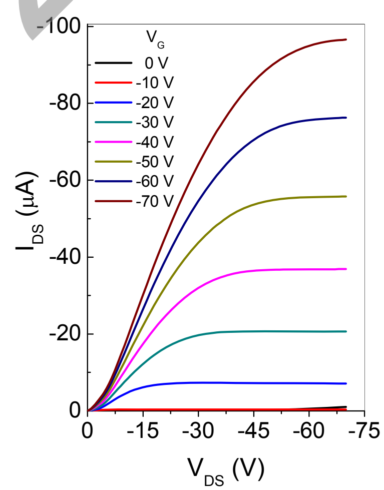



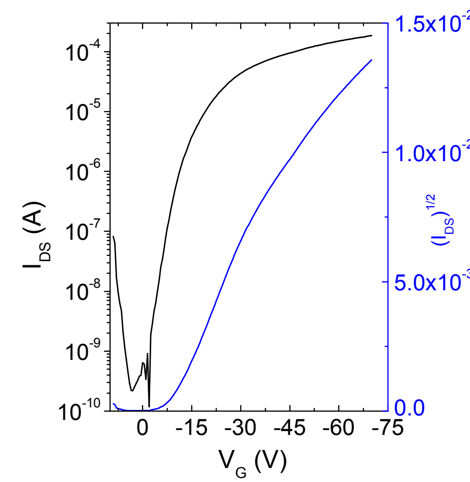

Output (left, $V_G$ = 0 V to -70V) and transfer (right, $V_{DS}$ = -70 V) characteristics of an OTFT device with a PDBT-co-TT thin film without annealing (*L* = 100 μm; *W* = 1 mm).

$\mu_h$= ~0.72 $cm^2$/V.s
$I_{on}/I_{off}$ = ~$10^{5\text{-}6}$
$V_T$ = -22 V

## Annealed at 200 °C

Output (left: $V_G$ = 0 V to -70 V) and transfer (right: $V_{DS}$ = -70 V) characteristics of an OTFT device with PDBT-co-TT thin film annealed at 200 ° C for 15 min. Device dimensions: channel length (L) = 100 μm; channel width (W) = 1 mm.

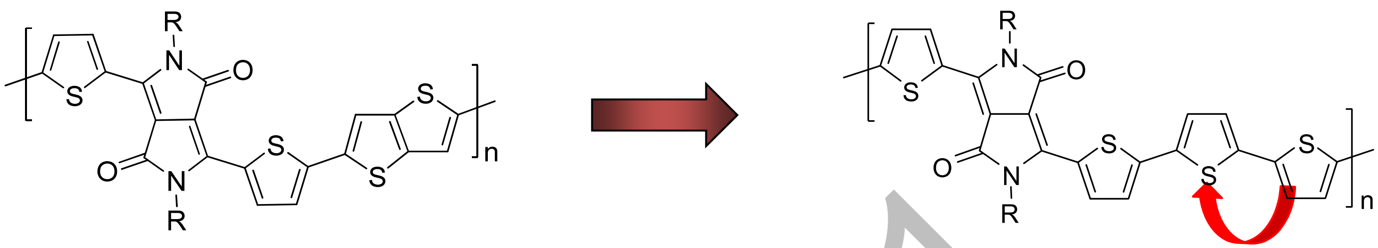


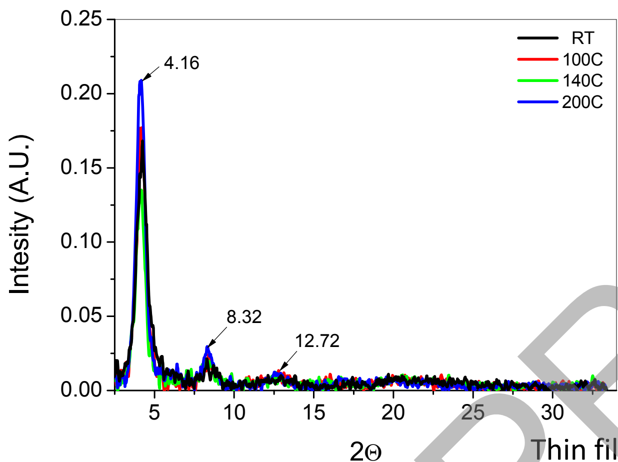

$\mu_h$= ~0.94 $cm^2$/V.s
$I_{on}/I_{off}$ = ~$10^{5\text{-}6}$
$V_T$ = -9 V

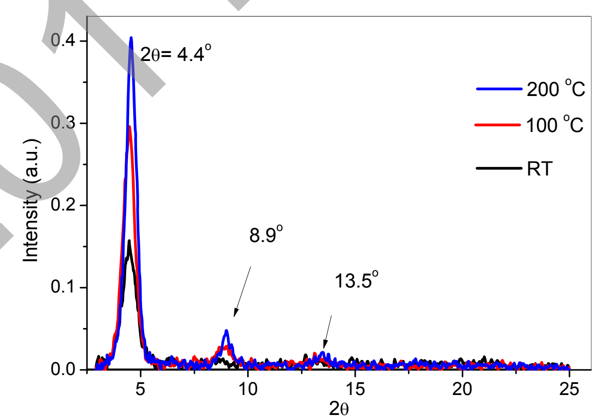
Y. Li, S. P. Singh, P. Sonar, *Adv. Mater*. 2010, *22*, 4862.

# PDQT

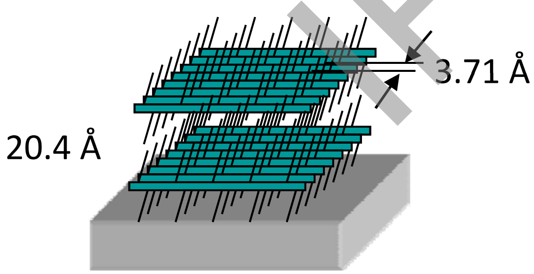


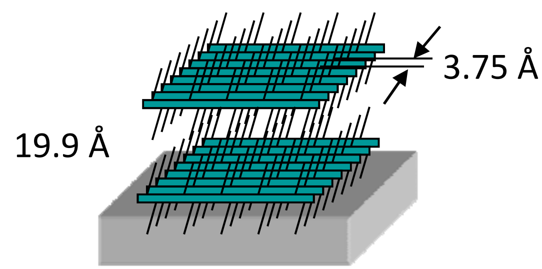

PDBT-co-TT PDQT




Thin film XRD data of DPP-LT thin films



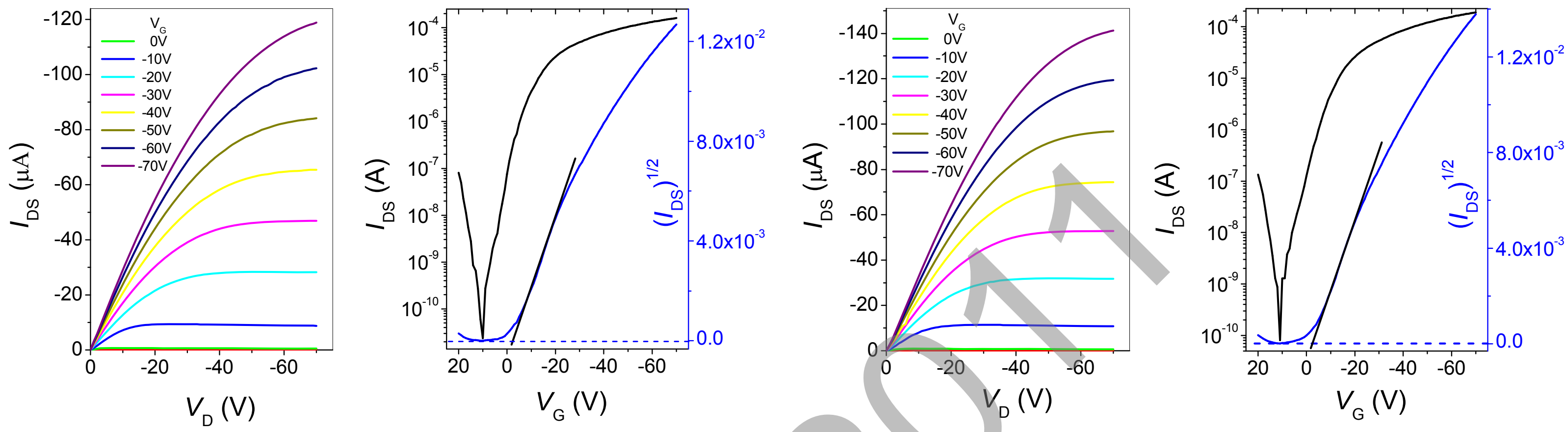




- Improved crystallinity by annealing
- Larger π-π distance and band gap

Li, Y.; Sonar, P.; Singh, S. P.; Soh, M. S.; van Meurs, M.; Tan, J. *J. Am. Chem. Soc.* **2011**, *133*, 2198.

# OTFT performance of PDQT






(a) (b) (c) (d)

As-span 100 ° C

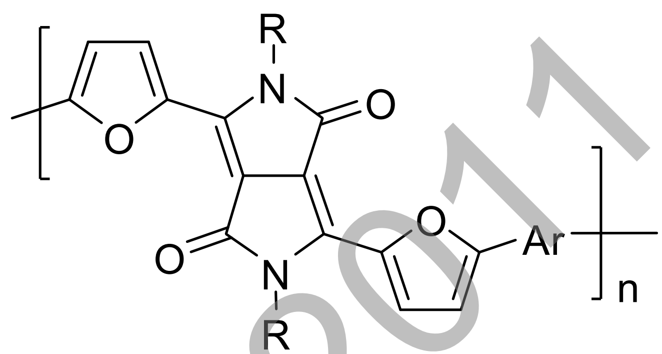
Output ($V_G$ = 0 V to -70V) and transfer ($V_D$ = -70 V) characteristics of OTFTs with DPP-LT thin films without annealing (a, b) and annealed at 100 ºC (c, d) ($L$ = 100 μm; $W$ = 1 mm).



| | **Hole mobility $\mu_h$, cm$^2$/V.s** | **Current on-to-off $I_{ON}/I_{OFF}$** | **Threshold voltage $V_T$, V** |
|---|---|---|---|
| Without annealing | **0.89** | ~$10^7$ | -3.0 |
| Annealed at 100 ºC/10min | **0.97** | 2x$10^6$ | -3.0 |

- Mobility is not very sensitive to annealing temperature (or crystallinity)
- Suitable for high-throughput roll-to-roll manufacturing

# Furan-DPP Polymers






- Furan is more electron-rich than thiophene
- Furan may suppress electron transport behaviour and make the hole transport more pronounced

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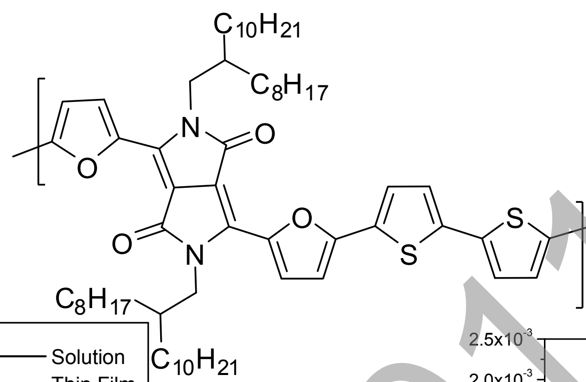
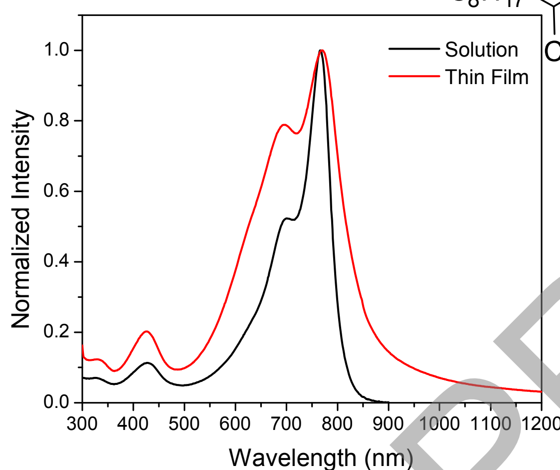
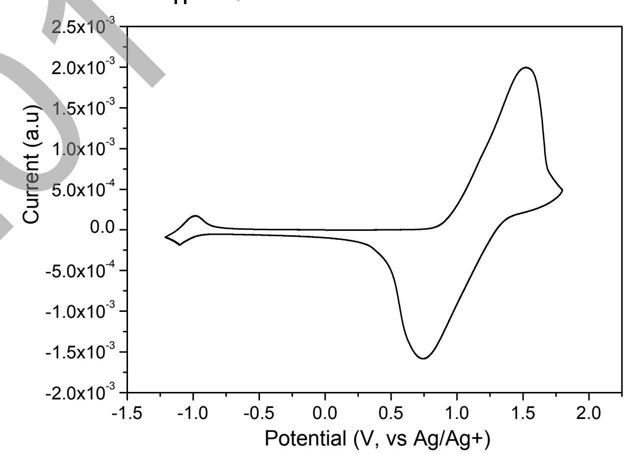
# PDBFBT

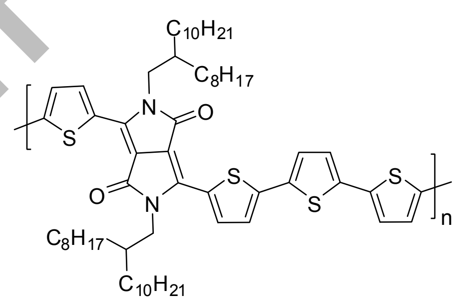



Eg = 1.41 eV

HOMO = 5.32 eV



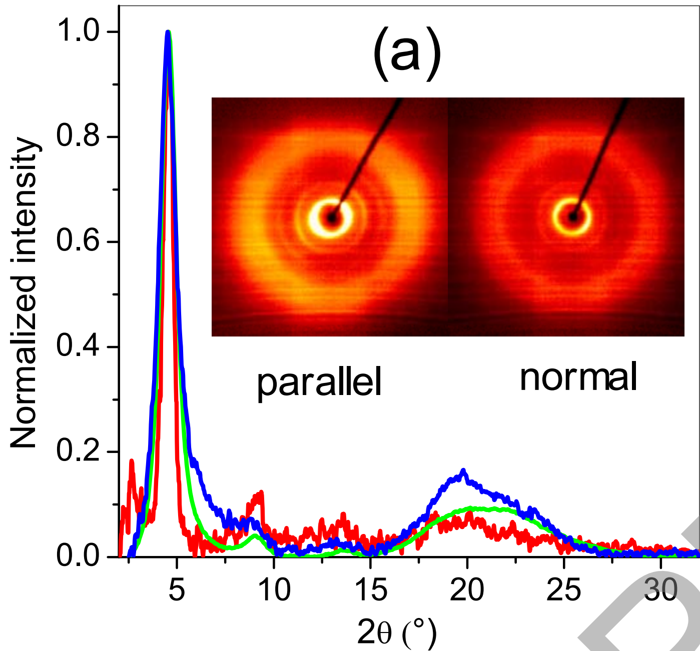

**PDQT**

$E_g$ =1.24 eV)

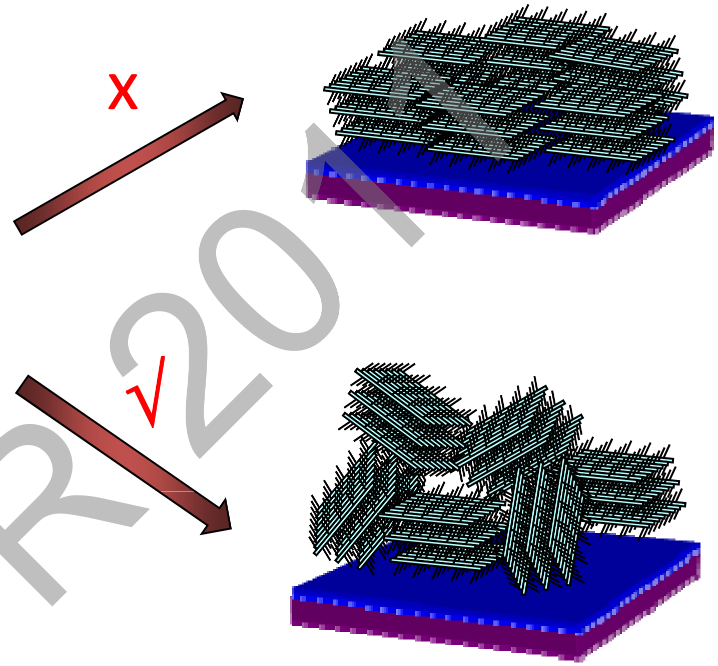
$E_{HOMO}$ = 5.20 eV

- Incorporation of furan broadens band gap and lowers HOMO level

# Molecular packing




1-D: Parallel to Spin-coated film
2-D XRD: Parallel to films
2-D XRD: Normal to films



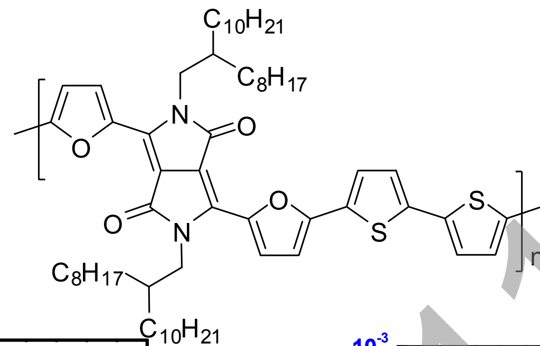

Edge-on

Random

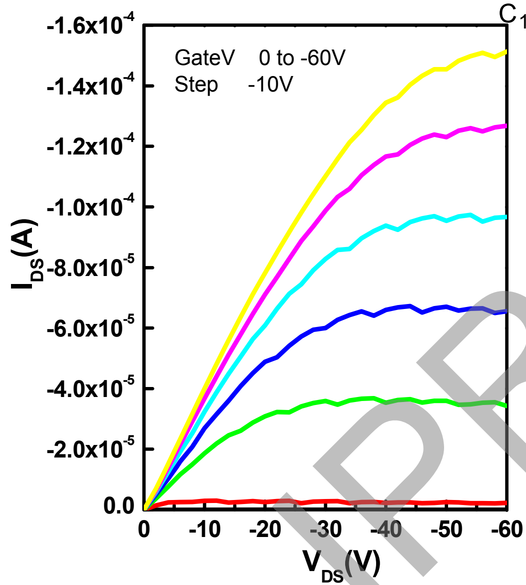


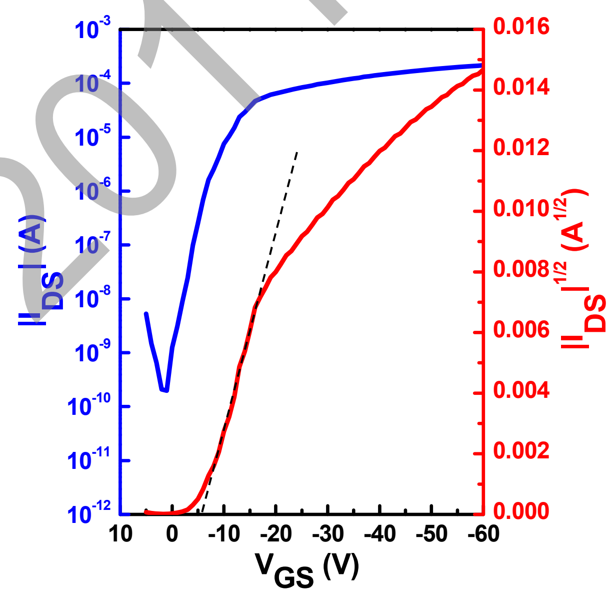
A mixture of edge-on and face-on orientations

# OTFT performance




PDBFBT



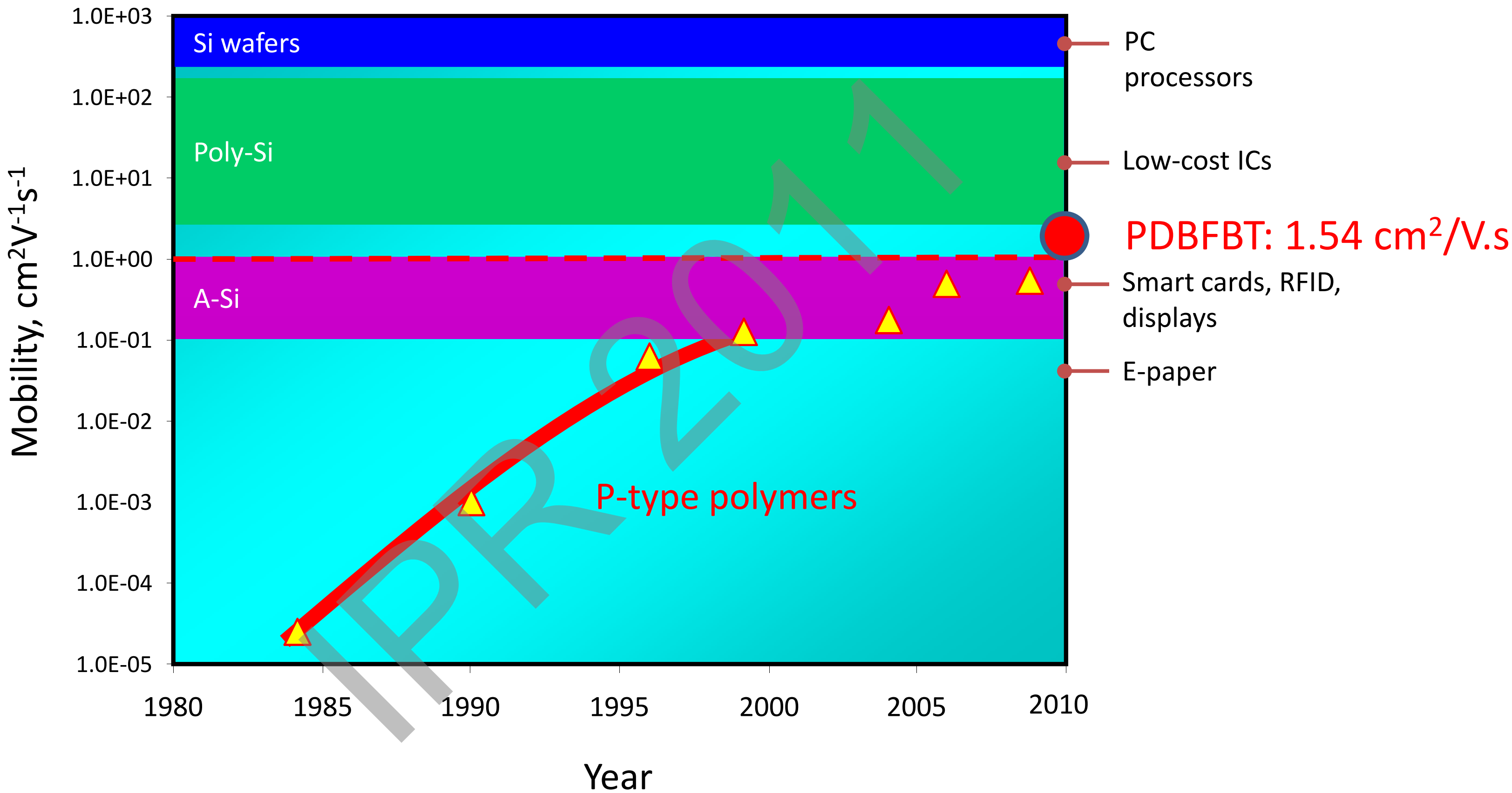



Output and Transfer characteristics of DA p-type polymer based OTFT (gold S/D; L = 125 µm; W = 4 mm) on OTS treated n+-Si/$SiO_2$ substrate (annealed at 200 ºC for 15 min)

Li,Y.; Sonar, P.; S. P. Singh, W. Zeng, M. S. Soh, *J. Mater. Chem*. under revision.

- $\mu_h$= ~**1.54 cm²/V.s** (annealed at 200 °C)
- $I_{on}/I_{off}$ = ~$10^{5\text{-}6}$

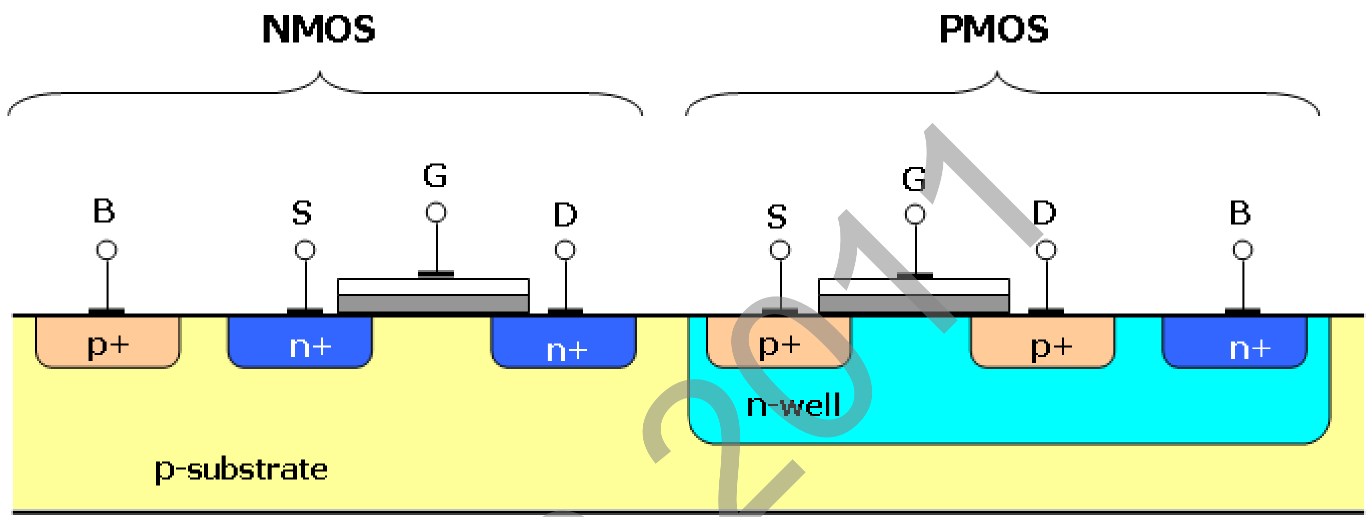
# Mobility of p-type polymer semiconductors




- Mobility of 1 cm$^2$/V.s is not an upper limit for polymer semiconductors
- Polymer semiconductors should show higher mobility than small molecules (record mobility: ~20 cm$^2$/V.s for single crystal rubrene)

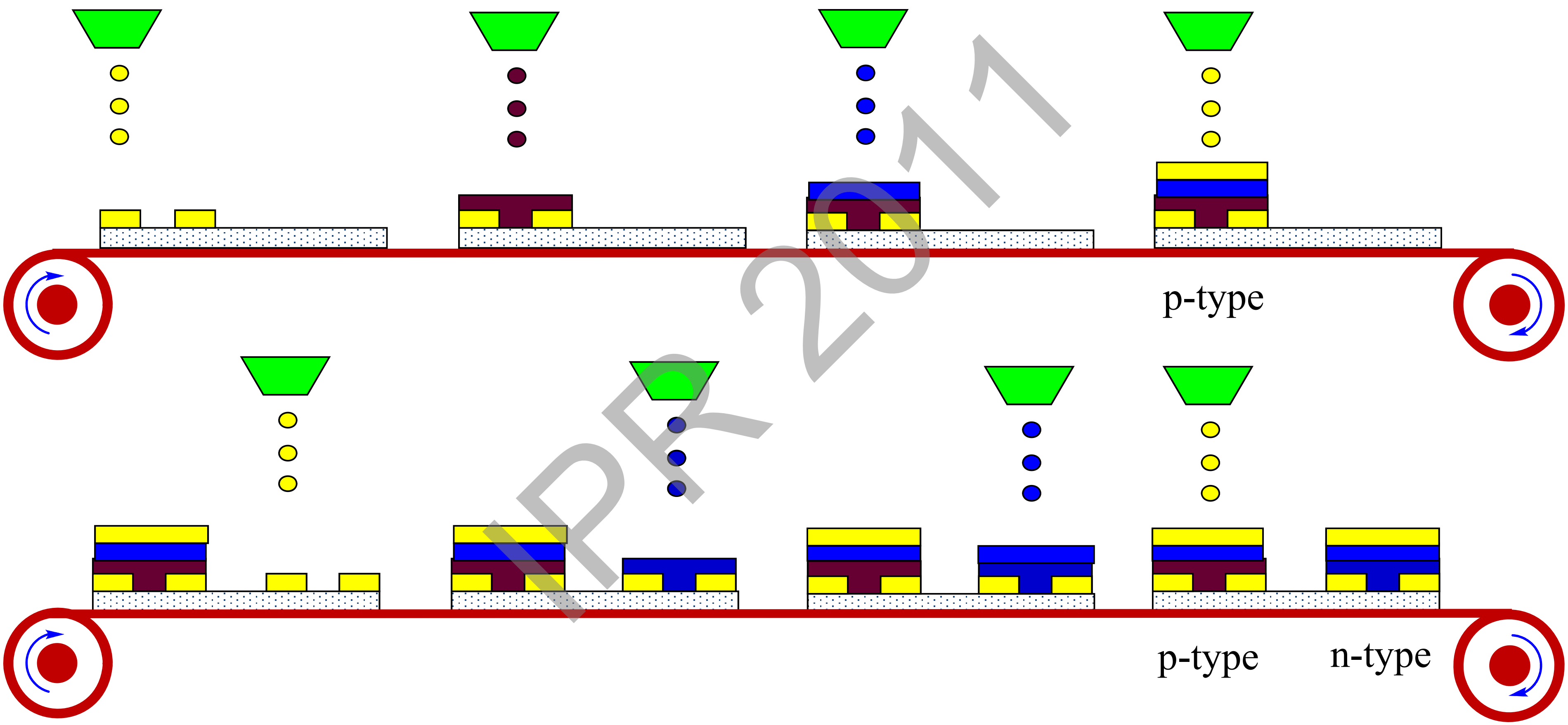
# Design of ambipolar polymer semiconductors for CMOS-like logic circuits

# CMOS (Complementary Metal Oxide Semiconductor)




- High robustness
- Lower power dissipation
- Higher noise immunity
- CMOS is dominating the integrated digital logic circuits

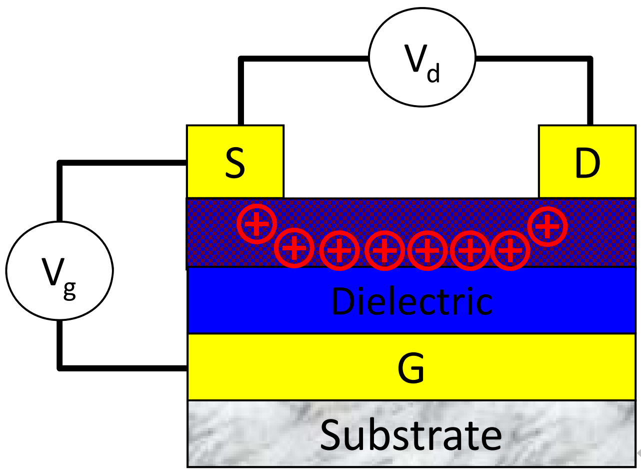
# Printing CMOS-like circuit with discrete p- and n-channel OTFT




- Complex fabrication process
- Difficulty in printing closely located p-type and n-type transistors

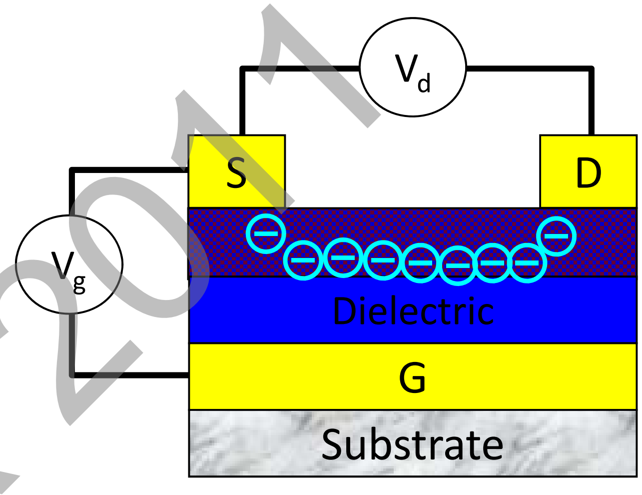
# Ambipolar polymer OTFT

P-type mode




$|V_g| > |V_d|$ , $V_g < 0$

N-type mode




$|V_g| > |V_d|$ , $V_g > 0$

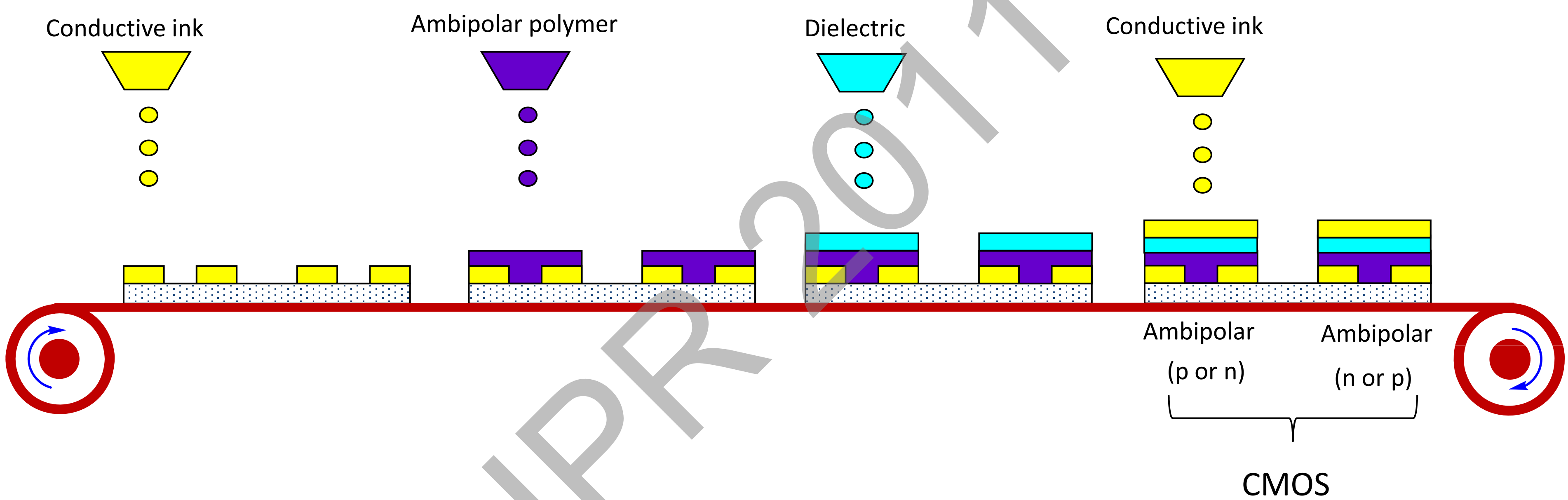
Meijer, et al, *Nat. Mater*. 2003, 2, 678 (Philips)
Bürgi, *et al, Adv. Mater.* 2008, *20*, 2217 (Ciba)



An ambipolar polymer can work as either a p-type semiconductor or an n-type semiconductor

# Printing CMOS-like circuits with ambipolar OTFT

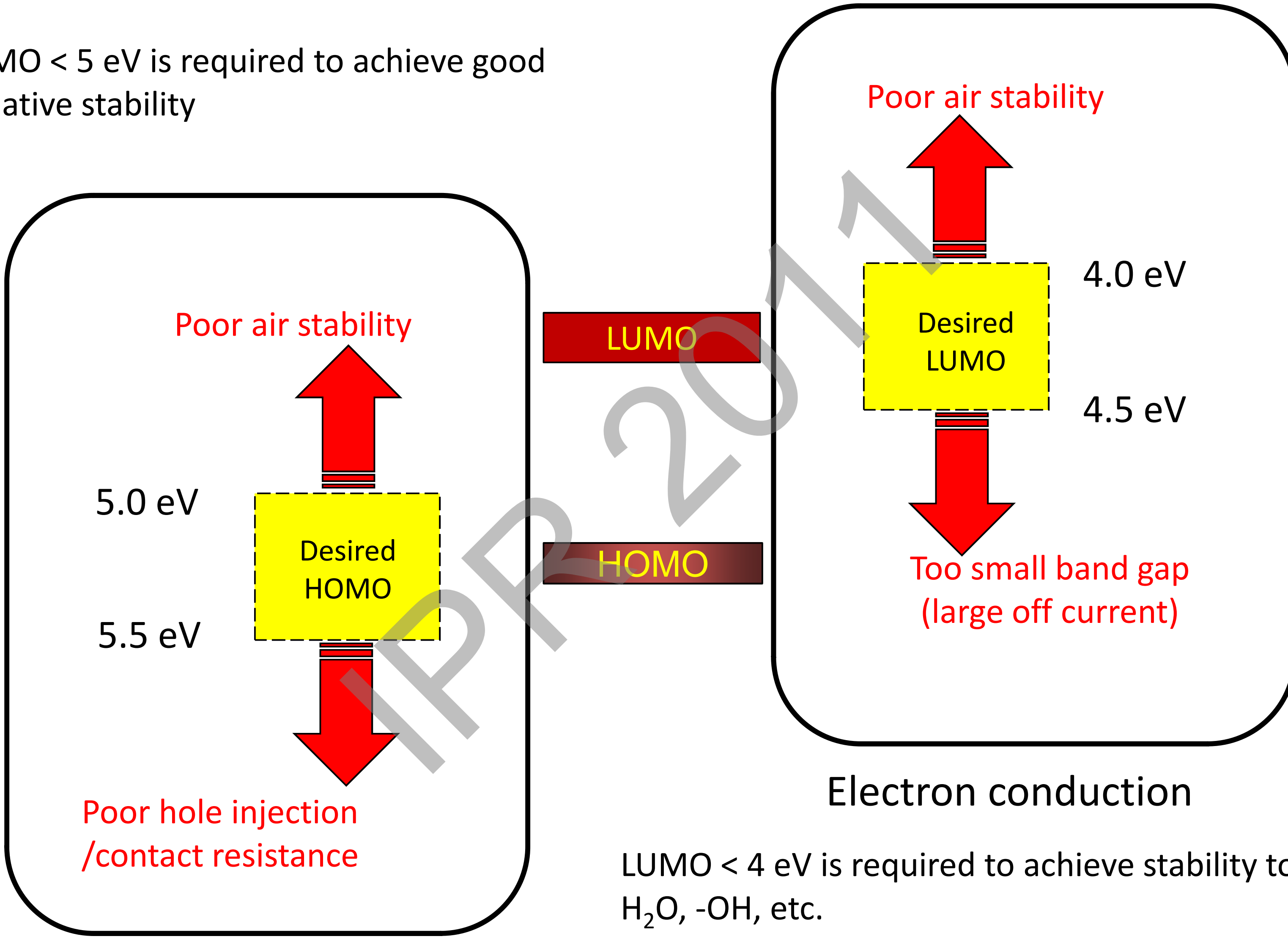





- Simplified printing process
- Improved device yield
- Reduced cost

# Ambipolar polymer design

HOMO < 5 eV is required to achieve good oxidative stability




Hole conduction

Electron conduction

LUMO < 4 eV is required to achieve stability towards $H_2O$, -OH, etc.

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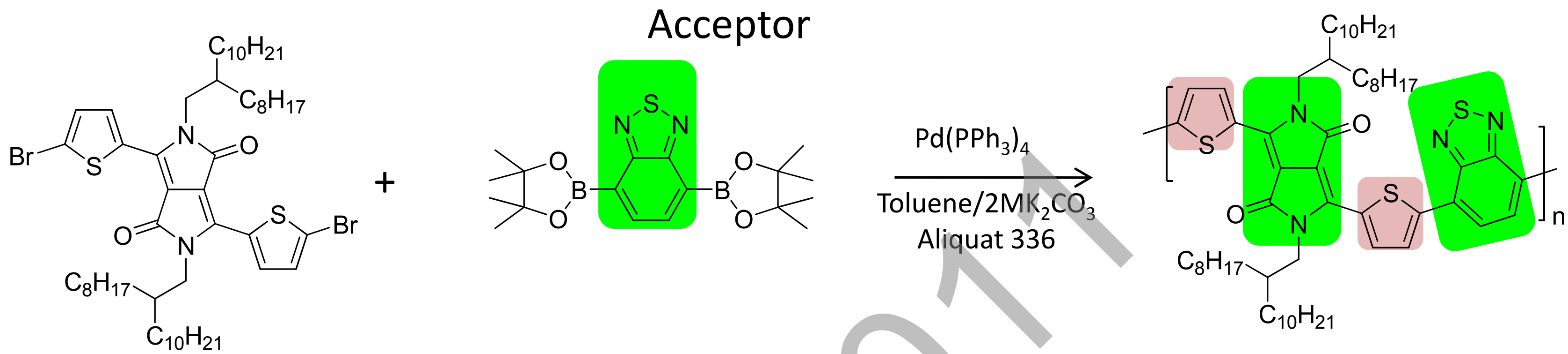
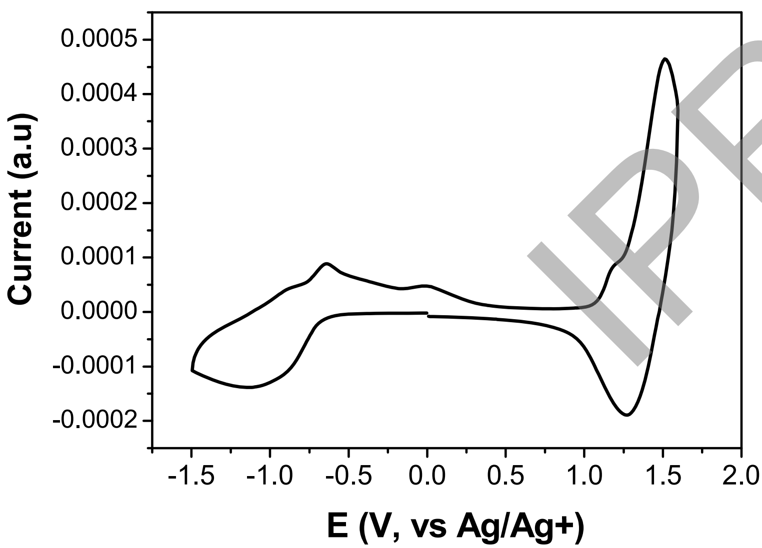
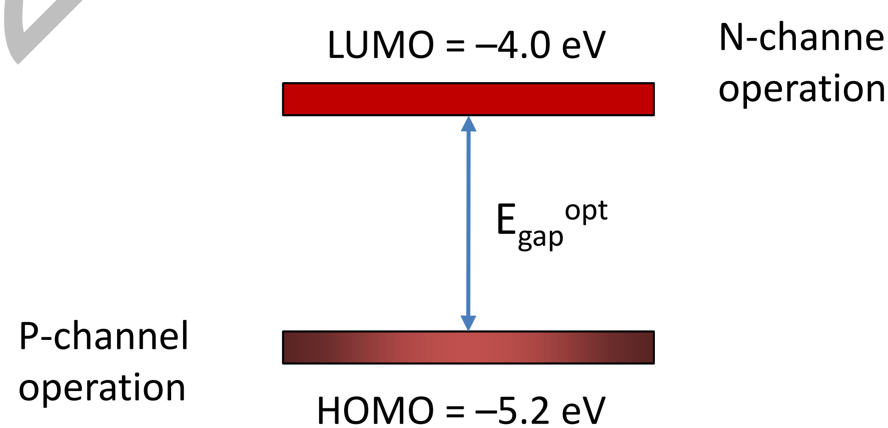
# PDPP-TBT ambipolar polymer



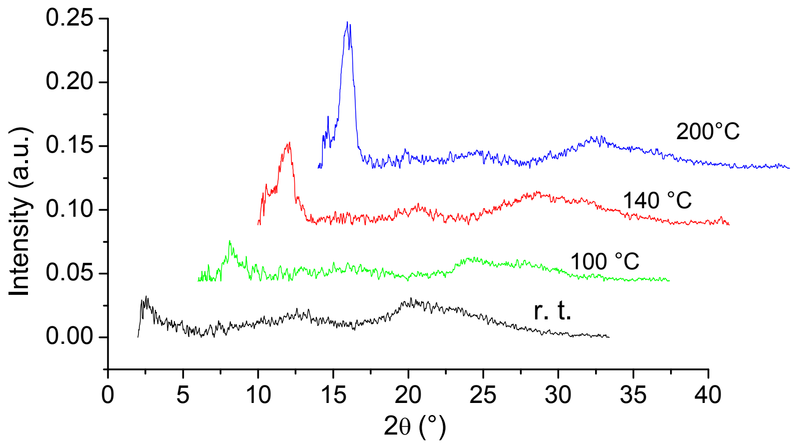

Sonar, P; Singh, S. P.; Li, Y.; Soh, M. S.; Dodabalapur, A. *Adv. Mater*. **2010**, *22*, 5409.



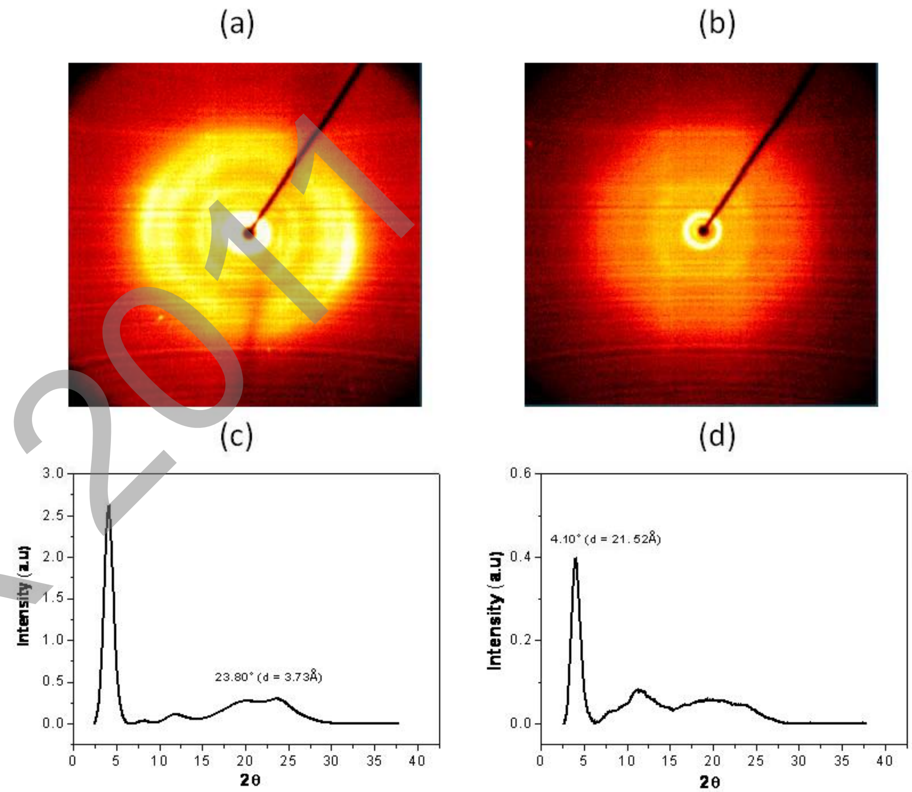




- Favored HOMO and LUMO levels for stable hole and electron transport

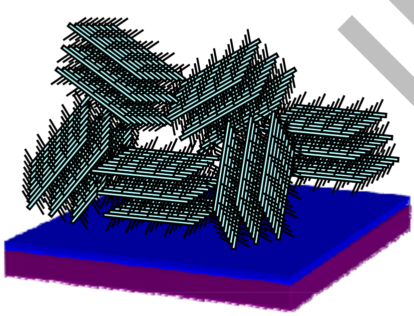
# Molecular organization




XRD data obtained from spin-coated PDPP-TBT thin films (~35 nm) on OTS modified $SiO_2$/Si substrates annealed at different temperatures.

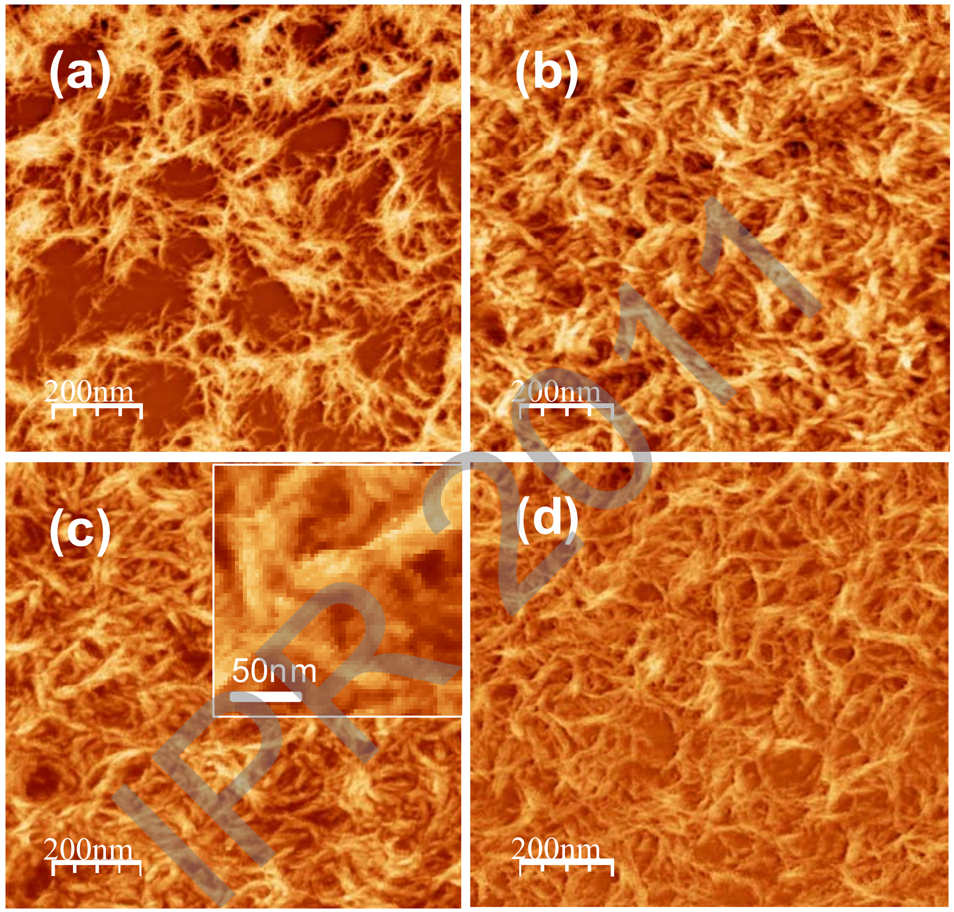



2-D XRD data for PDPP-TBT film stacks: (a) and (b) are, respectively, 2-D transmission XRD images obtained with the incident X-ray parallel and normal to the film stacks; (c) and (d) are, respectively, XRD diffractograms of pattern intensities of (a) and (b) obtained by integration of Chi (0-360° ) with GADDS software.



Randomly orientated?

# Thin film morphology



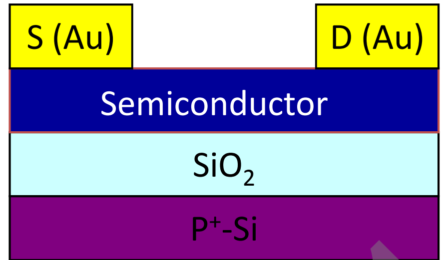


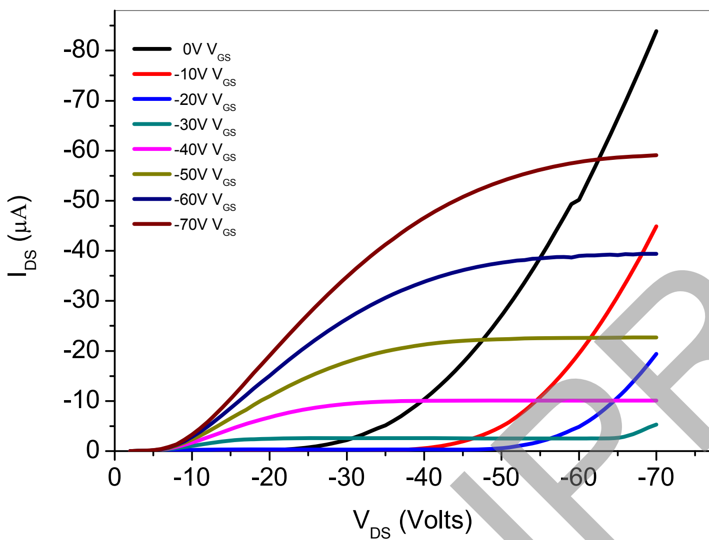

AFM phase images of PDPP-TBT thin films at: (a) room temperature, (b) annealed at 120° C, (c) annealed at 180° C, (d) annealed at 200° C on OTS treated $p^+$-Si/$SiO_2$ substrates. An inset zoom-in image in (c) shows clearly that each nanofiber is comprised of stacked nanorods.

- Crystalline fibrils grow as annealing temperature increases
- Intertwined networks facilitate interdomain charge transport

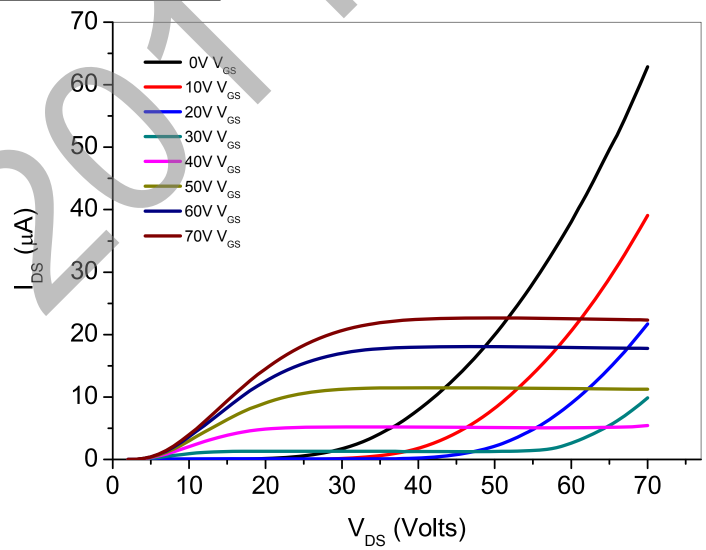
# OTFT performance of PDPP-TBT ambipolar polymer






Hole enhancement mode




Electron enhancement mode

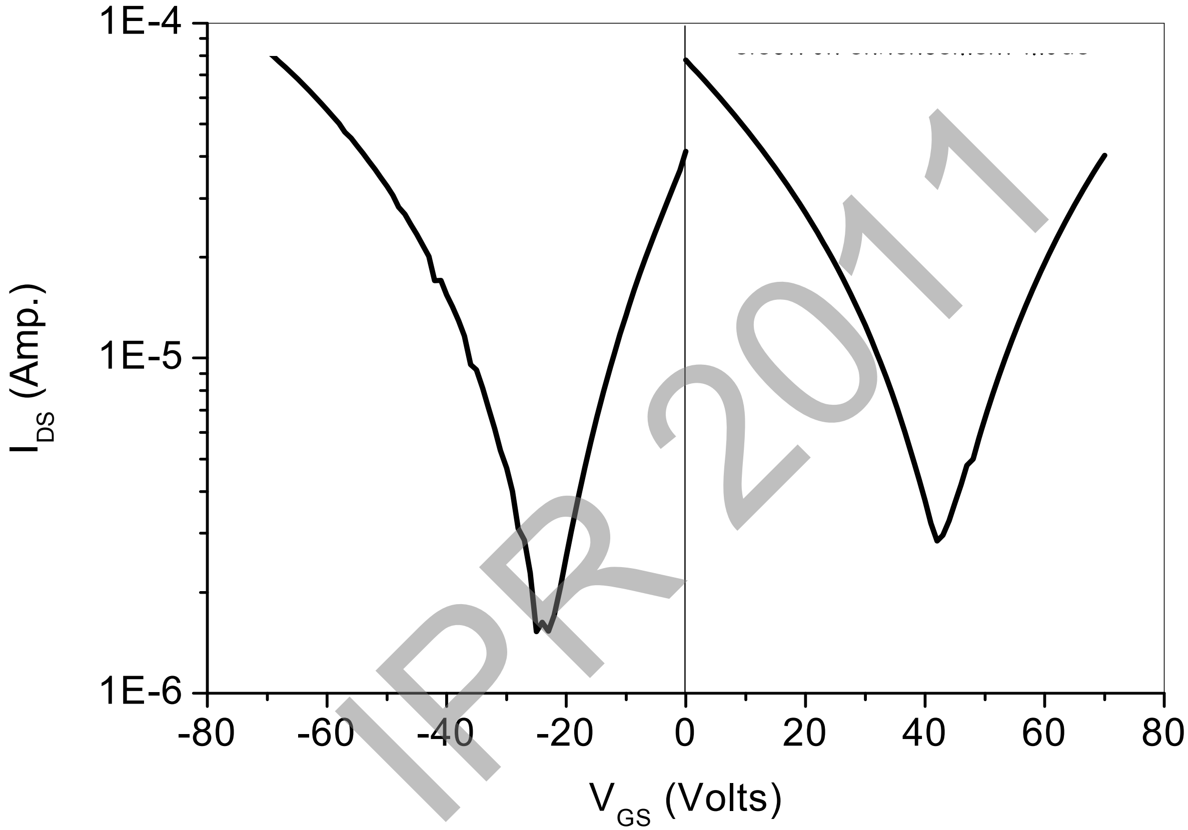


Output characteristics ($V_{DS}$ vs $I_{DS}$) of IMRE 1st Gen Ambipolar polymer based OTFT device annealed at 200 ° C on OTS treated p+-Si/$SiO_2$ substrate.

Sonar, P; Singh, S. P.; Li, Y.; Soh, M. S.; Dodabalapur, A. *Adv. Mater*. 2010, online

Characteristic behavior of an ambipolar OTFT

# OTFT performance of PDPP-TBT ambipolar polymer




Hole enhancement mode Electron enhancement mode

Transfer characteristics ($V_{GS}$-$I_{DS}$ ) OTFT device annealed at 200 ° C operated in hole (left) and electron (right) enhancement mode.

# OTFT performance of PDPP-TBT ambipolar polymer

Charge carrier mobility for PDPP-TBT Ambipolar polymer based OTFT

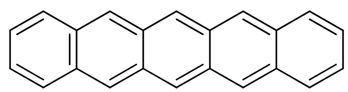
| Serial # | Annealing temperature, ° C | Charge carrier mobility ($cm^2$/V.s) | |
|---|---|---|---|
| | | Electron mobility ($\mu_e$) | Hole mobility ($\mu_h$) |
| 1 | Room temperature | 0.037 | 0.064 |
| 2 | 80 | 0.16 | 0.20 |
| 3 | 120 | 0.26 | 0.22 |
| 4 | 160 | 0.28 | 0.22 |
| 5 | 200 | **0.40** | **0.35** |

Sonar, P; Singh, S. P.; Li, Y.; Soh, M. S.; Dodabalapur, A. *Adv. Mater*. **2010**, *22*, 5409.



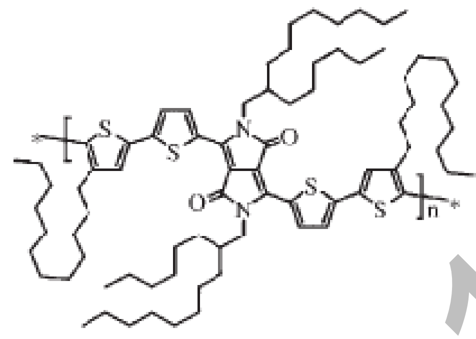
- Very well balanced, high electron and hole mobilities
- Excellent solubility/processability

# Comparison with other ambipolar polymers



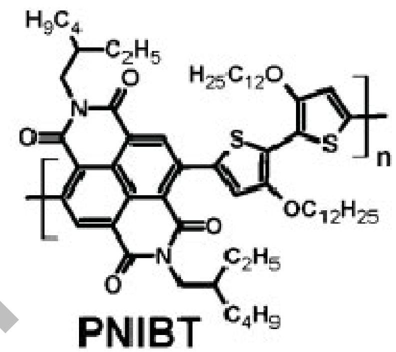
$\mu_e$ = 0.2 cm$^2$ /V.s
$\mu_h$ = 0.5 cm$^2$ /V.s
Au contacts
Vacuum deposition

Singh, *et al, Adv. Mater.* 2005, *17*, 2315.



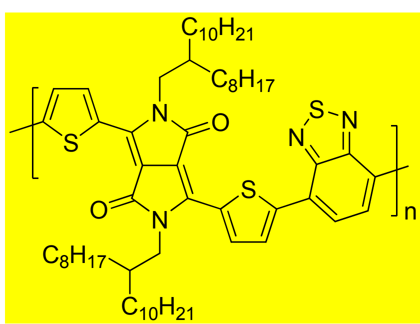

$\mu_e$ = 0.09 cm$^2$ /V.s
$\mu_h$ = 0.1 cm$^2$ /V.s
Ba contacts
Solution processed

Bürgi, *et al, Adv. Mater.* 2008, *20*, 2217.




$\mu_e$ = 0.04 cm$^2$ /V.s
$\mu_h$ = 0.003 cm$^2$ /V.s
Au contacts
Solution processed

Kim, *et, al, Adv. Mater*. 2010, *22*, 478.




PDPP-TBT ambipolar polymer
- $\mu_e$ = 0.40 cm$^2$ /V.s;
- $\mu_h$ = 0.35 cm$^2$ /V.s
- Au contact
- Solution processed

# Summary and Future Work

- Fused ring aromatic structures such as thienothiophene (TT) and benzodithiophene (BDT) could improve the charge carrier mobility up to 0.4 $cm^2/V.s$ due to increased π-π overlap and crystallinity.

- DPP-based polymers having intermolecular D-A interactions coupled with fused ring structures improved mobility up to 0.89 $cm^2/V.s$ and 1.54 $cm^2/V.s$ for respective annealing-free and annealed polymer thin films.

- By using appropriate design principles, ambipolar polymers with very balanced, high electron (0.40 $cm^2/V.s$) and hole mobility (0.35 $cm^2/V.s$) were developed, which are useful as one-component semiconductors for printed CMOS-like logic circuits.

- Currently working with Prof. Hany Aziz and Prof. William Wang in Electrical Engineering on printing OTFT arrays for OLED display applications.

- Aiming for polymers with high mobilities (~5-10 $cm^2/V.s$) for wider applications.

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*Thank you!*