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1. REPORT DATE (DD-MM-YYYY) 23-09-2012		2. REPORT TYPE FINAL		3. DATES COVERED (From - To) June 2009 to June 2012	
4. TITLE AND SUBTITLE Molecular Self-Assembly and Interfacial Engineering for Highly Efficient Organic Field Effect Transistors and Solar Cells				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER FA9550-09-1-0426	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Alex K.-Y. Jen				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Department of Materials Science and Engineering 302 Roberts Hall University of Washington				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Dr. Charles Y-C. Lee AFOSR/RSA 875 North Randolph Street Suite 325, Room 3112				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S) AFRL-OSR-VA-TR-2012-1126	
12. DISTRIBUTION/AVAILABILITY STATEMENT Distribution A APPROVED FOR PUBLIC RELEASE					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT Versatile surface and interface tailoring has been realized through molecular monolayers, polymer nanolayers or peptide monolayers. Insulating and semiconducting molecular phosphonic acid (PA) self-assembled monolayers (SAMs) have been developed for applications in organic field-effect transistors (OFETs) for low-power, low-cost flexible electronics. Multifunctional SAMs on ultrathin metal oxides, such as hafnium oxide and aluminum oxide, are shown to enable (1) low-voltage (sub 2V) OFETs through dielectric and interface engineering on rigid and plastic substrates, (2) simultaneous one-component modification of source/drain and dielectric surfaces in bottom-contact OFETs, and (3) SAM-FETs based on molecular monolayer semiconductors. (4) Polystyrene (PS) nanolayer as interface for OFETs, (5) threshold voltage control in OFETs with dielectric layer modified by the genetically engineered polypeptide (GEP) and (6) graphene oxide (GO) nanosheet-based OFETs and one diode-one resistor cell arrays for non-volatile memory have been also achieved. (7) Exquisite control of molecules is reached by monolayer assembly/confi					
15. SUBJECT TERMS Interface, Organic Field-Effect Transistors					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Alex K.-Y. Jen
U	U	U	U		19b. TELEPHONE NUMBER (Include area code) 206-543-2626

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Final Report

To: technicalreports@afosr.af.mil
Subject: Final Report to Dr. Charles Lee

PI: Alex K.-Y. Jen

Contract/Grant Title: Molecular Self-Assembly and Interfacial Engineering for Highly Efficient Organic Field Effect Transistors and Solar Cells

Contract/Grant #: FA9550-09-1-0426

Reporting Period: 6/1/09 – 6/30/12

Executive Summary:

Versatile surface and interface tailoring has been realized through molecular monolayers, polymer nanolayers or peptide monolayers. Insulating and semiconducting molecular phosphonic acid (PA) self-assembled monolayers (SAMs) have been developed for applications in organic field-effect transistors (OFETs) for low-power, low-cost flexible electronics. Multifunctional SAMs on ultrathin metal oxides, such as hafnium oxide and aluminum oxide, are shown to enable (1) low-voltage (sub 2V) OFETs through dielectric and interface engineering on rigid and plastic substrates, (2) simultaneous one-component modification of source/drain and dielectric surfaces in bottom-contact OFETs, and (3) SAM-FETs based on molecular monolayer semiconductors. The combination of excellent dielectric and interfacial properties results in high-performance OFETs with low-subthreshold slopes down to 75 mV/dec, high $I_{\text{on}}/I_{\text{off}}$ ratios of 10^5 - 10^7 , contact resistance down to 700 Ω cm, charge carrier mobilities of 0.1-4.6 $\text{cm}^2/(\text{V s})$, and general applicability to solution-processed and vacuum-deposited n-type and p-type organic and polymer semiconductors. (4) Polystyrene (PS) nanolayer as interface for OFETs, (5) threshold voltage control in OFETs with dielectric layer modified by the genetically engineered polypeptide (GEP) and (6) graphene oxide (GO) nanosheet-based OFETs and one diode-one resistor cell arrays for non-volatile memory have been also achieved. (7) Exquisite control of molecules is reached by monolayer assembly/confinement, and monitored by photon-STM for molecular motions, reactions and conductance in ground state and excited state.

1. SAM dielectric and interface engineering on rigid and plastic substrates

To produce low-voltage operating OFETs, there have been numerous attempts to develop gate dielectrics with increased capacitance density (C_i); this has been accomplished by either decreasing the thickness (d) or increasing the dielectric constant (k) ($C_i = \epsilon_0 k/d$). Common dielectric materials for low-voltage devices include ultrathin or high- k polymer films, polyelectrolytes, inorganic oxides, and hybrid organic/inorganic dielectrics. Molecular self-assembled monolayers and multilayers have also been proven to be excellent candidates for gate dielectrics in low-voltage OFETs. SAM dielectrics are composed of densely packed organic molecular monolayers that suppress carrier tunneling via highly ordered aliphatic chains even though they are only a few nanometer in thickness. In addition, by tuning the surface terminal group of the SAM, it is possible to modify the interface between the organic semiconductor and dielectric by exploiting compatible organic/organic interactions resulting in improved device performance. In the past few years, we have developed π - σ -PA SAMs on metal oxides such as HfO_x and AlO_x with the combination of excellent dielectric and interfacial properties to enable

low-voltage (sub 2V) high-performance OFETs based on solution-processed and vacuum-deposited n-type and p-type organic and polymer semiconductors (Fig. 1). Despite Al or Si gate electrode used in our work, these SAM/metal oxide hybrid dielectrics are compatible with other metal gate electrodes even printed ones and top-gate device architectures.

Ultrathin (<10 nm) hybrids of PA SAMs and metal oxides such as hafnium oxide and aluminum oxide have exhibited the combination of excellent dielectric and interfacial properties for low-voltage (sub 2V) high-performance OTFTs on rigid and flexible substrates. For pentacene OFETs on alkylphosphonic acid (σ -PA)-modified HfO_2 dielectrics, high saturation field effect mobilities result at a balance between disordered SAMs to promote large pentacene grains and thick SAMs to aid in physically buffering the charge carriers in pentacene from the adverse effects of the underlying high- k oxide. Compared to methyl-terminated σ -PA SAM/ HfO_2 (AlO_x) hybrid dielectrics, aryl-terminated π - σ -PA SAM/ HfO_2 (AlO_x) ones show improved dielectric properties such as leakage current density (down to $6 \times 10^{-9} \text{ A/cm}^2$ at 1.5 V) and capacitance density (up to $0.76 \mu\text{F/cm}^2$), and enhanced pentacene OFET device performance such as higher charge carrier mobility, current on/off ratio, and lower threshold voltage and subthreshold slope. This is due to more suppressing of carrier tunneling via ordered and longer molecular SAMs, better capacitive coupling of more polarizable aryl terminal group, and chemically and electrically more compatibility of π - σ -PA SAMs and organic semiconductors compared to σ -PA SAMs. The dielectric system of π - σ -PA SAM on low-temperature (<200 °C) processed HfO_x allow the fabrication of low-voltage TIPS-Pen and polymer OTFTs on flexible substrates, which represents a major advancement towards developing solution processed, low-power and flexible organic electronic devices. An all-additive patterning approach for SAM/ AlO_x hybrid dielectrics on Si substrates has been established to provide exceptional dielectric properties and compatible surface energy for subsequent patterning of solution processed n -channel and p -channel low-voltage OTFTs. These results provide a potential route for processing high-throughput, patterned, solution processed OTFTs for low-power electronic applications. Future design and synthesis of novel π - σ -PA SAMs on ultrathin metal oxides will allow interfacial interaction-induced optimization of orientation, packing and morphology of organic semiconductors towards low-voltage OTFTs with much enhanced device performance such as charge carrier mobility.

Low-V thin film transistors:

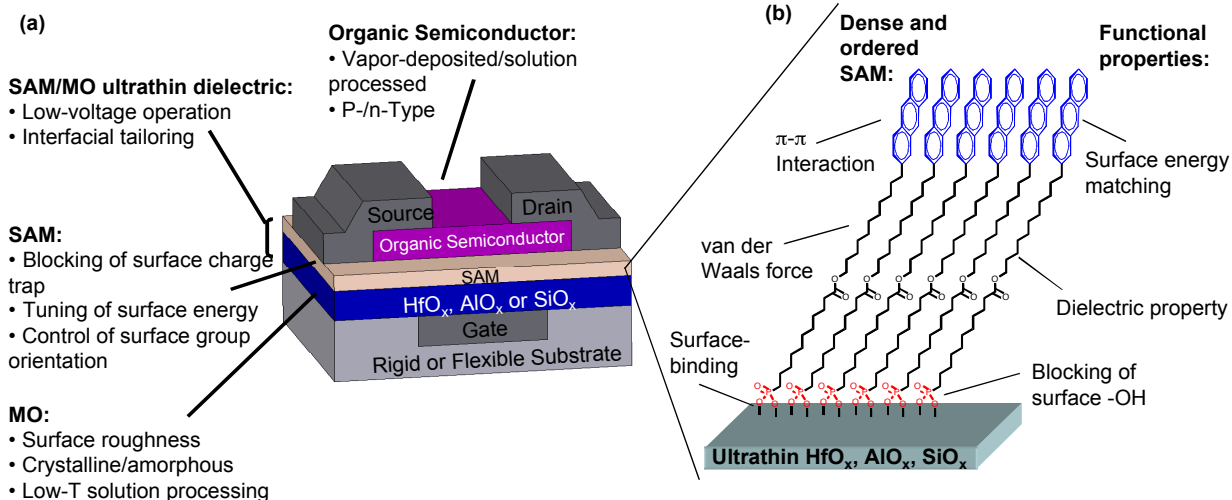


Fig. 1 (a) Schematic of SAM/metal oxide hybrids as ultrathin dielectrics and interface for low-voltage OFETs. (b) Typical π - σ -PA SAM on metal oxides with optimized dielectric and interfacial properties.

2. Simultaneous one-component SAM modification of source/drain and dielectric surfaces in bottom-contact OFETs

Approaches using SAMs to modify the electrodes in bottom-contact OTFTs are technologically important because bottom-contact devices are easily scalable by photolithography or printing which makes it a leading candidate for large-scale manufacturing. For tuning the metal/organic interface, SAMs with thiol as the binding group have been used to 1) tune the work function of the metal to match the energy level of organic semiconductor (OSC) to achieve Ohmic contact, 2) improve the order of the OSC by selective chemical SAM/OSC interactions, and/or 3) lower the surface energy of the metal surface. For the OSC/dielectric interface, SAMs with a wide range of binding groups (silanes, phosphonic acids, carboxylic acids, etc.) have been employed to control device performance. This has been achieved by, 1) passivating trap sites at the dielectric surface (e.g., -OH groups, and/or dielectric ionic polarization), 2) improving the order and/or charge accumulation in the OSC through chemical SAM/OSC interactions, and/or 3) matching the surface energy between the dielectric surface and the OSC. However, the commonly used methods for modifying bottom-contact electrode and dielectric surfaces in OTFTs have relied upon different processes with materials sometimes incompatible with each other. In addition, typical processing routes for high-quality SAM modification of metal electrode or dielectric surfaces require multiple steps and/or time consuming processing conditions (~10-48 h) from a combination of solution phase assembly, and extensive post processing (such as heating or vapor annealing). Therefore, it is imperative to develop a simplified method that can modify both bottom-contact electrode and dielectric surfaces simultaneously while maintaining desirable interfacial properties for high-performance OTFTs.

We have developed a simple and efficient process to simultaneously modify the OSC/bottom-contact electrode and OSC/dielectric interfaces with a single-component molecular SAM for bottom-contact OTFTs (Fig. 2). Using spin-coated OPA on Ag bottom-contact electrodes and ultra-thin HfO₂ dielectric, C₆₀ and pentacene based OTFTs can operate under 3 V with low contact resistance (down to 700 Ohm cm), low subthreshold swing (down to 75 mV/dec), high on-off current ratios of 10⁷, and charge carrier mobilities as high as 4.6 and 0.8 cm²/(V s) for C₆₀ and pentacene, respectively. Rational selection of the alkyl chain length of the SAM leads to optimized OTFT performance through a balanced combination of low-contact resistance at the bottom-contact electrodes and excellent interface properties for compact OSC grain growth. Future development of new π - σ -PA SAMs will allow simultaneous single-component modification of source/drain and dielectric surfaces to realize low-voltage high-performance bottom-contact OTFTs based on solution processed organic semiconductors.

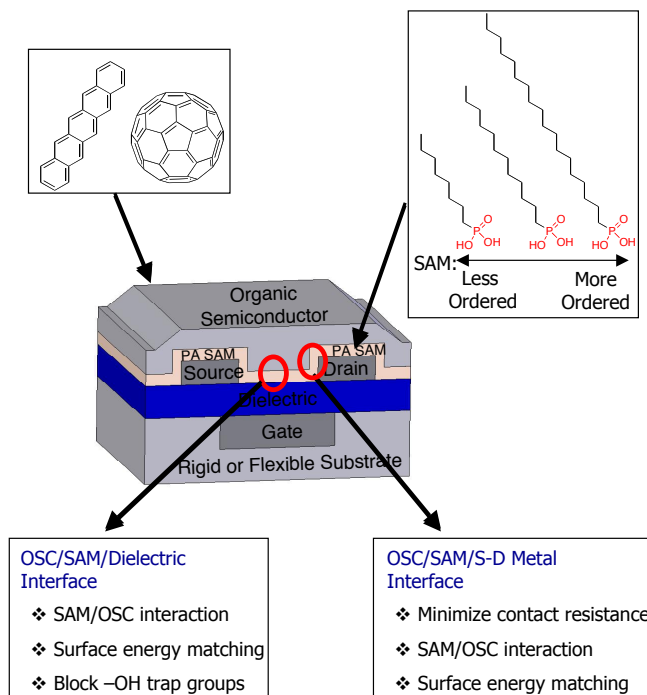


Fig. 2 Schematic of bottom-contact OTFTs using single-component spin-cast SAMs to simultaneously modify metal contacts and dielectric surfaces. Chemical structures of molecules used for SAMs in this study: *n*-octylphosphonic acid (OPA), *n*-dodecylphosphonic acid (DDPA), *n*-octadecylphosphonic acid (ODPA).

3. SAM-FETs based on molecular monolayer semiconductors

As it has been demonstrated that charge transport in OFETs primarily occurs in the first few nanometers of the semiconductor channel closest to the dielectric interface, therefore, devices utilizing a single molecular layer of semiconductor for charge transport are attainable. It has been recently realized that a molecular monolayer of organic semiconductor is sufficient to function as an ideal channel of an OFET with charge carrier mobility of up to $1 \text{ cm}^2/(\text{V s})$. This has inspired the development of π -conjugated semiconducting SAM molecules for fabricating self-assembled monolayer field effect transistor (SAM-FET). Functionally, SAM-FETs are OFETs in which the organic semiconductor is a single layer of well-packed π -conjugated molecules capable of acting as a charge-transporting channel. Recently, SAM-FETs with long channel lengths of up to $40 \text{ }\mu\text{m}$ have been demonstrated, in which a SAM of oligothiophene was formed on a SiO_2 or polymeric gate dielectric via a greater than 15 h of immersion assembly. Due to long-range intermolecular π - π coupling in the monolayer, performance of these SAM-FETs was comparable to that of OFETs constructed with a thin bulk film. However, for all the SAM-FETs fabricated so far, most devices required channel lengths of submicrometer to ensure a gate voltage modulation of the source-to-drain channel current. This is because it is difficult to achieve fast charge transport due to defects and poor π - π coupling between molecules in such long channels.

We have recently demonstrated rapidly processed SAM-FETs achieved through spin-coating a phosphonic acid-based molecule 11-(5'''-butyl-[2,2';5',2";5'',2''';5''',2''''']quinquethiophen-5-yl)undecylphosphonic acid (BQT-PA) (Fig. 3). The resulting spin-cast SAMs show uniform density, and well-ordered monolayer coverage

comparable to that observed by conventional immersion phase solution assembly. The top-contact SAM-FETs processed by spin-coating and immersion assembly show identical electronic performance. These devices were found to have an average μ of $1.1 - 8.0 \times 10^{-6} \text{ cm}^2/(\text{V s})$ for a wide range of channel lengths (device channel lengths measured were 12, 20, 30, 50 and 80 μm). On/off current ratio ($I_{\text{on}}/I_{\text{off}}$) for most devices was on the order of 10^2 .

Complementary to OTFTs, SAM-FETs are versatile and promising because layers of dielectrics, semiconductors, and electrode contact can be tailored in a molecular level to combine ultrathin monolayer-metal oxide hybrid dielectrics, monolayer semiconductor with controlled molecular orientation and packing, and chemically bonded electrode contact in a single system. The strategy to self-assemble multifunctional molecules with semiconducting, dielectric and surface-binding moieties onto ultrathin metal oxides such as AlO_x and HfO_x is highly attractive because it can enhance the understanding of the dependence of charge mobility on molecular structure, orientation, and packing. This will help advance OFET technology and realize its full potential for low power consumption, versatile bottom-up processing, and ultimate miniaturization.

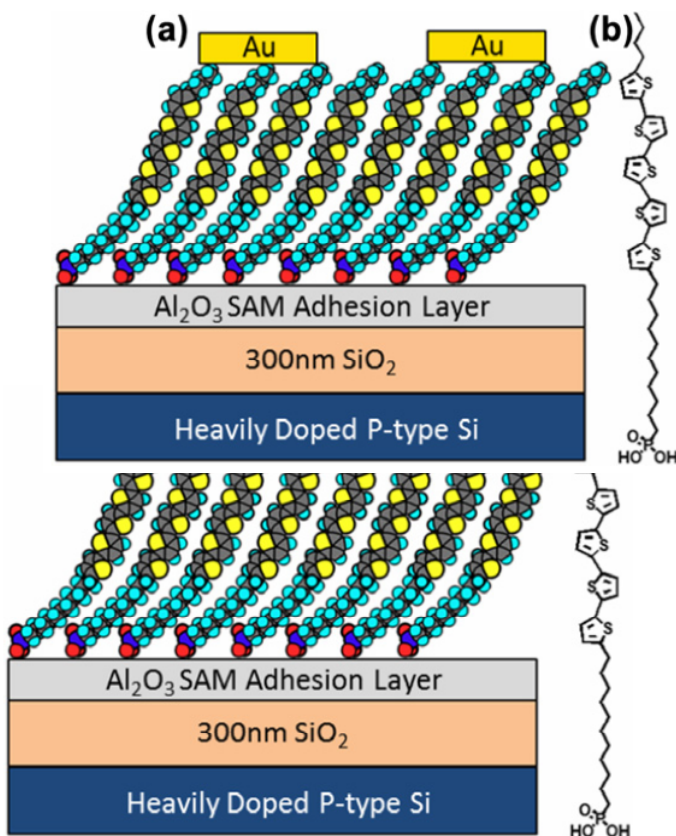


Fig. 3 (a) SAM-FET device architecture (cartoon). From bottom: P-doped Si with 300 nm oxide, 2 nm adhesion layer, BQT-PA, gold electrodes. (b) Chemical structure of BQT-PA.

4. Polystyrene (PS) nanolayer as interface

The PS films were inserted between SiO₂ and organic semiconductors as buffer layers for OFETs. The results showed that a flat orientation of phenyl ring at the surface of the PS films optimized the surface energy of PS film, resulting in higher crystallinity of pentacene films deposited onto it and an improved interconnection between the pentacene crystalline domains. The device with the PS film thermally treated at 120 °C showed superior performance, affording a mobility as high as 4 cm²/V s, an on/off ratio of about 10⁷-10⁸ and a threshold voltage of about 6.5 V in the saturation region.

Low-voltage pentacene-based OFETs were also demonstrated with PS/HfO_x hybrid dielectrics. Thermal annealing the PS at 120 °C readily improves the quality of the hybrid dielectric surface by inducing flatter phenyl group orientation and better matched surface energy with pentacene compared to untreated PS. Pentacene deposited on PS-120 display higher quality thin films with larger grain sizes and higher crystallinity. Pentacene OFETs with PS-120/HfO_x hybrid dielectrics can operate at low-voltage (< 3 V) with high field-effect mobilities (1 cm²/V s), high on/off current ratios (10⁶), and low subthreshold slopes (100 mV/dec).

5. Threshold voltage control in OFETs with dielectric layer modified by the genetically engineered polypeptide (GEP).

Important device parameters for OFETs include driving voltage, stability, on/off current ratio, charge carrier mobility, and threshold voltage (V_T). Controlling V_T can yield higher circuit performance and lower power consumption in electronics. It can be shifted by modifying the dielectric with self assembled monolayers (SAMs) to shift V_T by tens of volts.

We have achieved precise control over the threshold voltage of pentacene-based organic thin-film transistors by inserting a genetically engineered quartz-binding polypeptide at the semiconductor-dielectric interface. A 30-V range was accessed with the same peptide by adjusting the pH of the solution for peptide assembly while leaving other device properties unaffected. Mobility of 0.1-0.2 cm² V⁻¹ s⁻¹ and on/off current ratio of >10⁶ could be achieved for all devices regardless of the presence of the neutral peptide or the peptide assembled in acidic or basic conditions. This shift of threshold voltages is explained by the generation of charged species and dipoles due to variation in assembling conditions. Controlling device characteristics such as threshold voltage is essential for integration of transistors into electronic circuits.

6. Graphene oxide (GO) nanosheet-based OFETs and one diode-one resistor cell arrays for non-volatile memory

We have demonstrated ONVMTs using chemically synthesized and spin-coated GO nanosheets as the charge-trapping layer. Based on the applied gate bias, the transfer curve characteristics showed positive or negative threshold voltage shifts, indicating charge trapping or detrapping in GO nanosheets. It exhibited more than two orders of ON/OFF ratio for 10⁴ s. Although the memory performance needs to be further improved, GO based ONVMTs may be promising for the next generation of non-volatile memory applications.

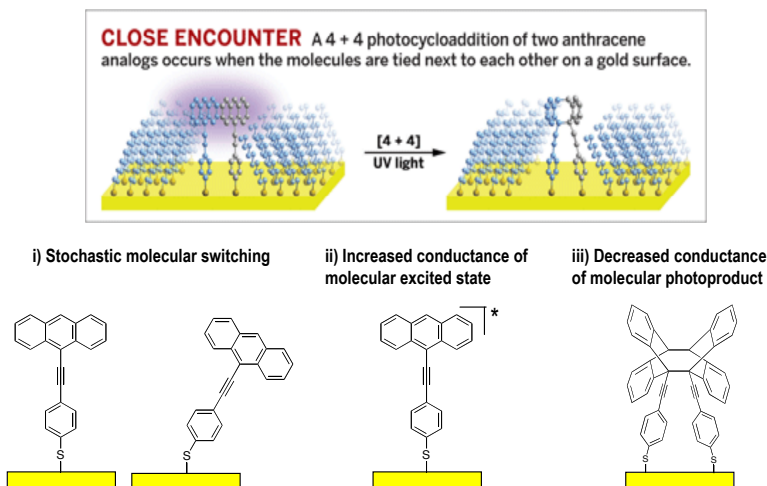
We have also introduced photolithographically patterned organic resistor and diode materials, and successfully fabricated and tested the performance of a prototype organic photopatterned 4 x 4 1D-1R cell array. Photopatterning of the resistor layer does not affect the cell operation (e.g. sweep endurance and retention properties) compared to the previously reported device performance. In addition, after the integration of photopatterned organic schottky

diodes on top of the photopatterned 1R cells, the 1D–1R cell exhibited improved reading accessibility and memory properties, while eliminating cross-talk with neighbouring cells. These results demonstrate the feasibility of fabricating high-density, active-type, all-organic memory 1D–1R cell arrays and generating organic electronic devices with sophisticated architectures.

7. STM investigation of photoactive SAMs with confined environment and controlled orientation under light irradiation

Exquisite control of molecules is reached by monolayer assembly/confinement, and monitored by photon-STM for molecular motions, reactions and conductance in ground state and excited state. Three distinct phenomena on anthracene-terminated phenylethynyl thiolates molecules on Au{111} were observed including stochastic switching, increased conductance for molecules in excited states, and drops in conductance attributed to photochemical reactions. Exercising control over molecular reactivity by confinement and STM monitoring molecular motions and reactions have great potential for both understanding and measuring complex chemical reactions. Fundamental understanding of molecules in ground state, excited state, light absorption, charge separation, charge generation and charge transport on surface is crucial for organic electronics, including organic transistors, solar cells and light-emitting diodes.

Confined Surface for Photoreaction and Molecular Electronics



Jen, et al. *Science* **2011**, 331, 1312-1315.

Archival publications (published) during reporting period:

1. Ma, H., Acton, O., Hutchins, D. O., Cernetic, N., Jen, A. K.-Y., “Multifunctional Phosphonic Acid Self-Assembled Monolayers on Metal Oxides as Dielectrics, Interface Modification Layers and Semiconductors for Low-Voltage High-Performance Organic Field-Effect Transistors”, *Phys. Chem. Chem. Phys.* **2012**, DOI: 10.1039/C2CP41557G. (**Featured on Front Cover**).
2. Hutchins, D. O., Acton, O., Weidner, T., Cernetic, N., Baio, J. E., Ting, G., Castner, D. G., Ma, H., Jen, A. K.-Y., “Solid-State Densification of Spun-Cast Self-Assembled Monolayers

for Use in Ultra-Thin Hybrid Dielectrics”, *Appl. Surface Sci.* **2012**, <http://dx.doi.org/10.1016/j.apsusc.2012.09.013>.

3. Kim, T. W., Zeigler, D. F., Acton, O., Yip, H. L., Ma, H., Jen, A. K.-Y., “All-Organic Photopatterned One Diode-One Resistor Cell Array for Advanced Organic Nonvolatile Memory Applications”, *Adv. Mater.* **2012**, *24*, 828. **(Featured on Front Cover)**.
4. Hutchins, D., Acton, O., Weidner, T., Cernetic, N., Baio, J. E., Ting, G., Castner, D. G., Ma, H., Jen, A. K.-Y., “Spin Cast Self-Assembled Monolayer Field Effect Transistors”, *Org. Ele.* **2012**, *13*, 464.
5. Acton, O., Hutchins, D., Arnadottir, L., Weidner, T., Ting, G. G., Kim, T. W., Cernetic, N., Castner, D. G., Ma, H., Jen, A. K.-Y., “Spin-Cast and Patterned Organo-Phosphonate Self-Assembled Monolayer Dielectrics on Metal Oxide Activated Si”, *Adv. Mater.* **2011**, *23*, 1899-1902.
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7. Kim, M., Hohman, J. N., Cao, Y., Houk, K. N., Ma, H., Jen, A. K.-Y., Weiss, P. S., “Creating Favorable Geometries for Directing Organic Photoreactions in Alkanethiolate Monolayers”, *Science* **2011**, *331*, 1312-1315. **(Highlighted on Chem. & Eng. News, March 14, 2011. Highlighted by UW Today, March 15, 2011. Highlighted by UCLA Newsroom, March 10, 2011.)**
8. Kim, T. W., Gao, Y., Acton, O., Yip, H. L., Ma, H., Chen, H. Z., Jen, A. K.-Y., “Graphene Oxide Nanosheets Based Organic Field Effect Transistor for Non-Volatile Memory Applications”, *Appl. Phys. Lett.* **2010**, *97*, 023310.
9. Dezieck, A., Acton, O., Leong, K., Oren, E. E., Ma, H., Tamerler, C., Sarikaya, M., Jen, A. K.-Y., “Threshold Voltage Control in Organic Thin Film Transistors with Dielectric Layer Modified by the Genetically Engineered Polypeptide”, *Appl. Phys. Lett.* **2010**, *97*, 013307.
10. Wang, Y., Acton, O., Ting, G., Weidner, T., Shamberge, P. J., Ma, H., Ohuchi, F. S., Castner, D. G., Jen, A. K.-Y., “Effect of the Phenyl Ring Orientation in the Polystyrene Buffer Layer on the Performance of Pentacene Thin-Film Transistors”, *Org. Ele.* **2010**, *11*, 1066-1073.
11. Ma, H., Yip, H.-L., Huang, F., Jen, A. K.-Y., “Interface Engineering for Organic Electronics”, *Adv. Func. Mater.* **2010**, *20*, 1371-1388 **(invited review article)**.
12. Acton, O., Ting, G., Shamberger, P. J., Ohuchi, F. S., Ma, H., Jen, A. K.-Y., “Dielectric Surface Controlled Low-Voltage Organic Transistors via n-Alkyl Phosphonic Acid Self Assembled Monolayers on High-*k* Metal Oxide”, *Appl. Mater. Interface* **2010**, *2*, 511.
13. Wang, Y., Acton, O., Ting, G., Weidner, T., Shamberge, P. J., Ma, H., Ohuchi, F. S., Castner, D. G., Jen, A. K.-Y., “Effect of the Phenyl Ring Orientation in the Polystyrene Buffer Layer on the Performance of Pentacene Thin-Film Transistors”, *Org. Ele.* **2010**, *11*, 1066.
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15. Acton, O., Osaka, I., Ting, G., Hutchins, D., Ma, H., McCullough, R. D., Jen, A. K.-Y., “Phosphonic Acid Self-Assembled Monolayer and Amorphous Hafnium Oxide Hybrid

Dielectric for High Performance Polymer Thin Film Transistors on Plastic Substrates”, *Appl. Phys. Lett.* **2009**, 95, 113305.

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17. Ma, H., Liu, M. S., Jen, A. K.-Y., “Interface-Tailored and Nanoengineered Polymeric Materials for (Opto)electronic Devices”, *Poly. International* **2009**, 58, 594. **(invited review article).**

Cumulative lists of people involved in and the research effort.

Orb Acton, graduate student, material processing and OFET characterization (Ph. D. obtained in 2010)

Dan Hutchins, graduate student, material processing and OFET characterization (M. S. obtained in 2012)

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Dr. Hong Ma, research assistant professor, material design, synthesis, and processing

Changes in research objective, if any: None

Changes in AFOSR program manager, if any: None

Extensions granted or milestones slipped, if any: None

Include any new discoveries, inventions, or patent disclosures during this reporting period (if none, report none):

Jen, A. K.-Y., Acton, O., Cernetic, N., Hutchins, D., Ma, H., “Rapidly Processed Electronic Self-Assembled Monolayers and Approaches Thereof”, US Application Number 61/486,921 (2011)