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MURI: Electrochemically-Controlled Orthogonal Assembly on Monolayers on a Gold Surface via DPN

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13. ABSTRACT *(Maximum 200 Words)*

Nature utilizes molecular recognition between complex macromolecules to form sophisticated meso- and macroscopic architectures with tremendous control over the placement and orientation of nanoscopic building blocks within the extended architectures. The structures of proteins, duplex DNA, and phospholipid bilayers are maintained by cumulative weak and reversible interactions (hydrogen bonding and van der Waals interactions), and pairing principles for the constituent molecules. The biosyntheses are remarkably efficient, self-correcting, and high yielding; therefore, they are excellent models for how the synthetic chemist should consider designing complex nanostructured materials. The specific objective of the proposed research was to demonstrate orthogonal assembly of charged particles by electrochemically controlling particle deposition. In addition, the use of applied potentials to selectively miniaturize microscopic features to nanoscopic dimensions was explored.

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14. SUBJECT TERMS
orthogonal assembly, electrochemical, particle deposition

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Air Force Office of Scientific Research

Final Report

for Grant: F49620-00-1-0345

"MURI: Electrochemically-Controlled Orthogonal Assembly of Monolayers on a Gold Surface via DPN,"

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October 28, 2003

Period covered: 08/01/00-07/31/03

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Final Report- "MURI: Electrochemically-Controlled Orthogonal Assembly of Monolayers on a Gold Surface via DPN"

Grant Number: F49620-00-1-0345

PI NAME: Chad A. Mirkin, Northwestern University

Period covered: 08/01/00-07/31/03

1. OBJECTIVES

2. STATUS OF WORK

We have shown that we can initiate and direct the assembly of polyanionic oligonucleotide-modified particles in an orthogonal manner using predesigned templates comprised of redox-active components on gold substrates. We generate the multicomponent structures via Dip-Pen Nanolithography (DPN) by utilizing alkanethiols with electroactive end groups as inks, e. g. $Fc(C{=}O)(CH_2)_{10}SH$ and $Fc(CH_2)_{11}SH$. The design and choice of inks allows us to successively oxidize the terminal ferrocenyl groups of the different inks in the pattern in order to bind negatively charged oligonucleotide-modified nanoparticles in an orthogonal manner. This novel approach not only provides a method for addressing individual nanostructures fabricated via DPN, but also offers a route to assembling complex, multicomponent structures on these redox-active templates.

We have also realized a simple and convenient strategy for reducing the dimensions of organic micro- and nanostructures on metal surfaces. By varying electrochemical desorption conditions, features consisting of linear alkanethiols or selenols patterned by Dip-Pen Nanolithography (DPN) or Micro-Contact Printing (μ-CP) can be gradually desorbed in a controlled fashion. The process is called electrochemical whittling because the adsorbate desorption is initiated at the exterior of the feature and moves inward as a function of time. The whittling process and adsorbate desorption were studied as a function of substrate morphology, adsorbate head and tail groups, and electrolyte solvent and salt. Importantly, one can independently address different nanostructures made of different adsorbates and effect their miniaturization based upon a judicious selection of adsorbate, applied potential, and supporting electrolyte. Some of the physical and chemical origins of these observations have been elucidated.

By taking advantage of the electrochemical whittling process, we have recently developed a methodology for fabricating metal nanostructures from DPN generated templates. This strategy takes advantage of the different desorption rates between alkylselenols and alkylthiols. Using DPN, we can generate a pattern consisting of alkylselenols and then passivate of the bare gold with alkylthiol. The alkylselenol can be selectively desorbed by applying the appropriate potential, thus leaving a bare gold template. This template can be used to electrochemically deposit metal salts to generate the desired metal nanostructures.

ACCOMPLISHMENTS/NEW FINDINGS

Redox-controlled orthogonal assembly of charged nanostructures. We have shown how one can use predesigned, redox-active ferrocenylalkylthiol "inks" ($Fc(CH_2)_{11}SH$ **(1)** and $Fc(C{=}O)(CH_{11})SH$ **(2)**), patterned on a gold substrate, and appropriately applied changes in electrode potential that result in ink oxidation, to trigger and guide the assembly of polyanionic oligonucleotide-modified particles in an orthogonal manner (Figure 1).

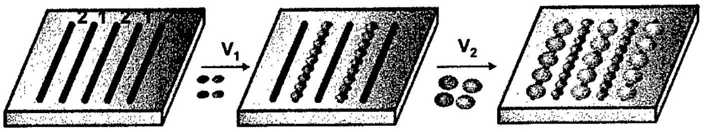

Figure 1. Schematic diagram of the assembly of charged nanostructures.

The ferrocenyl based inks were designed to exhibit nonoverlapping redox processes by virtue of the alkylthiol and acylthiol functional groups directly attached to the cyclopentadienyl rings in **1** and **2**, respectively. Indeed, cyclic voltammetry of gold electrodes modified with monolayers of **1** and **2**, respectively, exhibit a $\Delta E_{1/2}$ of 255 mV (Figure 2A). The rationale behind choosing these two molecules was that one could selectively address nanostructures made of **1** and then **2** by sweeping the potential of an electrode modified with them in linear fashion. The concomitant oxidation of the nanostructures would result in the selective deposition of oppositely charged species onto the nanostructure of interest (Figure 1). In the case of the system described, oligonucleotide modified nanoparticles were chosen as nanostructure building blocks for two reasons. First, they exhibit low non-specific adsorption on uncharged monolayer modified surfaces, and second, they provide a proof-of-concept system for demonstrating the suitability of this strategy for arranging complex, chemically tailorable, and functional nanostructured building blocks on a templated surface.

In a typical experiment, the redox-active inks are patterned by DPN on a gold surface to generate charge-neutral nanostructures (Figures 2B, 2C). The structures are generated in serial fashion from tips coated with the appropriate ink. Prior to imaging the nanostructures, a tip coated with 1-octadecanethiol (ODT) was raster scanned across the patterned area to passivate the exposed gold surface. Upon completion of all DPN writing steps each sample was rinsed with ethanol, further passivated by soaking the substrate in a 1 mM ethanol solution of ODT for 1 min, and rinsed one more time with ethanol.

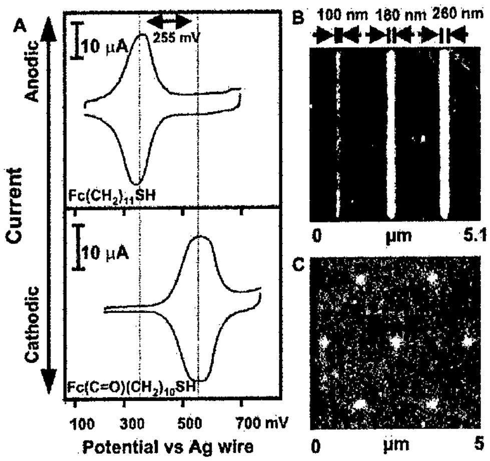

Figure 2. (A) CVs in 0.2 M Hcl04 of monolayers of **1** (top) and **2** (bottom) vs Ag/AgCl. (B) LFM image of DPN-generated lines of **1**. (C) LFM image of an array of dots of **2**.

Both **1** and **2** result in patterns, that exhibit higher lateral force (light) relative to the passivated gold substrates (dark) (Figures 2B, 2C). Thus far, we have generated features with linewidths as small as 55 nm and as large as 1 μm. The size of the pattern can be adjusted by changing the scan speed (line-based features) or tip-substrate contact time (dot-based arrays) during the writing procedure.

The orthogonal assembly of oligonucleotide functionalized nanoparticles was effected by using the patterned substrate as a working electrode in an electrochemical cell. To attach particles to substrates containing patterns drawn with only one ferrocenyl-based ink, the oxidation was done at room temperature using fixed pulse potentials at either 350 mV (for **1**) or 620 mV (for **2**) vs Ag/AgCl for 30 min in the presence of Au nanoparticle solutions in 0.15 M NaCl PBS buffer (10 mM phosphate buffer, pH 7). Gold nanoparticles, ~5 nm and 13 nm in diameter, were functionalized with alkylthiol-terminated DNA strands based on published protocols. In the case of samples containing patterns drawn with both ferrocenyl alkanethiolates, linear sweep voltammetry was used to address each pattern where the potential of the working electrode was ramped from 0 to 350 mV, held for 30 min in the presence of a solution of 5 nm Au particles, and then taken to 620 mV and held for 30 min in the presence of 13 nm particles. The cell and electrode were washed with 0.15 M NaCl PBS buffer between each step and at the end of the experiment. In addition, the substrates were rinsed with 0.05 % Tween 20 solution and nanopure water and cleaned with Scotch tape, as described by Sagiv et. al., prior to imaging. Subsequent imaging of the multicomponent two-dimensional arrays of particles was performed using a Nanoscope IIIa and microscope from Digital Instruments in tapping mode under ambient conditions with silicon cantilevers (spring constant = ~ 40 N/m).

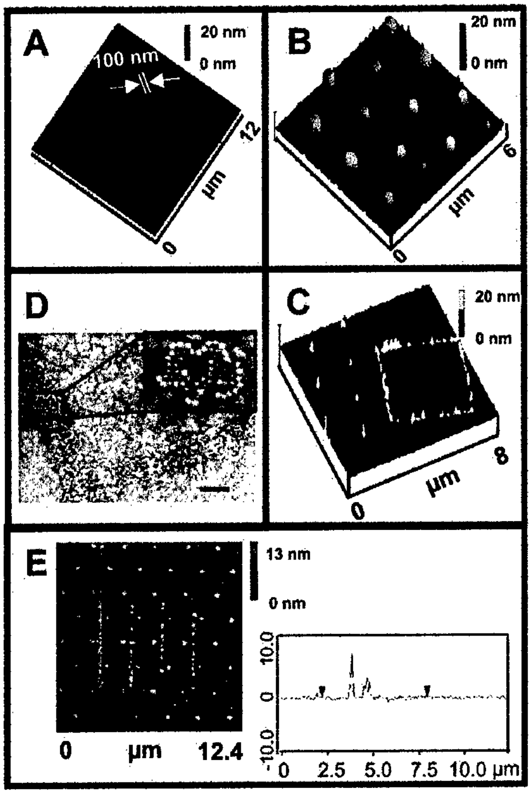

Figure 3. Three-dimensional nano-structures assembled onto electro-chemically active patterns. (A) Tapping mode image of 13 nm particles assembled onto features drawn with **1**. (B) Tapping mode image of 13 nm particles assembled onto features drawn with **2**. (C) Taping mode image of 13 nm particles assembled onto features drawn with **1** and **2**. (D) High-resolution field-emission scanning microscopy image of a portion of a square pattern after particle assembly. (E) Tapping mode image of 5 nm particles assembled onto line features drawn with **1** and 13 nm particles assembled onto dot patterns made from **2**.

Single ink structures, comprised of either **1** or **2** can be used to guide the assembly of 13 nm particles (Figures 3A, 3B). Note that the electrode washing steps and scotch tape are very effective at removing particles that stick to the passivated areas of the gold electrode but not the oxidized and positively charged nanostructures. A substrate with two patterns, one comprised of **1** (the square) and the other comprised of **2** (the dots) can be used to sequentially assemble 13 nm particles on the preformed templates via the linear sweep voltammetry protocol (Figure 3C). To verify that we have indeed assembled a high density array of Au nanoparticles onto the desired patterns with a low degree of non-specific binding, we obtained a high resolution, field-emission scanning electron microscopy (FE-SEM) image of the patterned substrate after the electrostatic assembly process (Figure 3D). The data clearly show that the ~13 nm particles are closely packed and localized predominantly on the template features. Finally, we were able to attach different sized oligonucleotide modified nanoparticles onto two different nanopatterns (a line array of **1** and a dot array of **2**) in an orthogonal manner using the linear sweep voltammetry protocol and 5 and 13 nm particles, respectively (Figure 3E). The height profile of the assembled structures confirms that 5 nm particles are present on the lines of **1** and the 13 nm particles are on the dot arrays drawn with **2**.

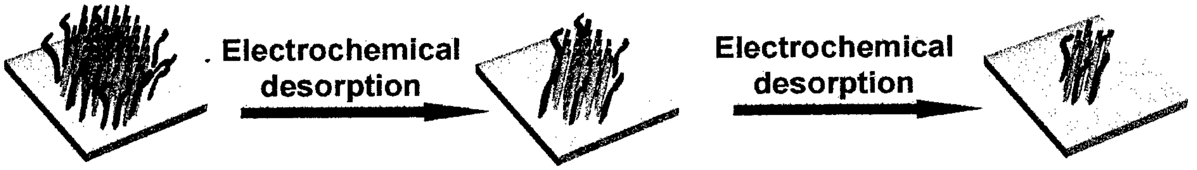

Figure 4. Alkanethiol patterns are whittled away from the edges using the defect sites at the perimeter.

Electrochemical whittling of organic nanostructures. We have recently realized a massively parallel strategy for reducing the size of DPN generated alkanethiol nanostructures on gold surfaces. This approach works by the electrochemical desorption of organic nanostructures (Figure 4). For example, by using a conventional three-electrode cell, we were able to reduce the size of MHA patterns from 90 nm to 55 nm by applying a potential of –750mV for 1 minute, effectively reducing the diameter of these structures at rate of ~1 molecule/sec (Figure 5). The periphery of nanostructures, which contains many defect sites are gradually whittled away at a rate dependant on the time and potential applied. Not only does this strategy provide a simple and convenient method to enhance the resolution of DPN, but also holds potential in improving the resolution of all soft lithography techniques. Also, by applying this procedure to structures patterned on atomically flat surfaces we should potentially be able to break the current 15 nm resolution limit of DPN.

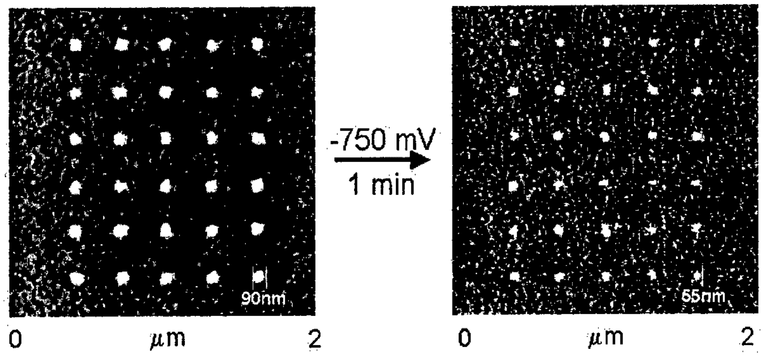

Figure 5. Lateral Force Microscopy (LFM) images of (A) original MHA dots array and (B) the MHA dots array after holding the potential at –750 mV for 1 min. The size of the dots in (A) have been reduced from ~90nm to ~55nm as can be seen in (B).

Two Component Whittling. Due to the different reduction potentials of MHA and ODT on gold surfaces, it is possible to selectively desorb nanostructures of one ink on the surface while leaving the second ink. As shown in Figure 6, it is possible to completely desorb MHA (light in LFM) and leave the ODT nanostructures intact (dark in LFM) by applying a voltage of –750 mV for 6 minutes.

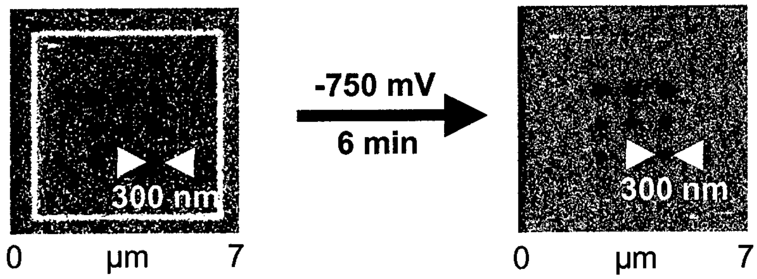

Figure 6. Lateral Force images before applying a -750 mV pote a substrate patterned with ODT and MHA. The MHA lines de e the ODT dots remain intact.

Parallel Size Reduction of Organic Microstructures. We have extended the electrochemical whittling method to organic structures fabricated by high-throughput parallel printing techniques. Indeed, this is where the utility of the technique could be realized for miniaturizing micro or relatively large nano-scale architectures in a massively parallel fashion. By printing 16-mercaptohexadecanoic acid (MHA) and 1-octadecanthiol (ODT), then applying a reducing potential in 0.5 M KOH, we show that it is possible to electrochemically whittle these structures (Figure 7). We have figured out the conditions to optimize this process and have begun to take advantage of its utility.

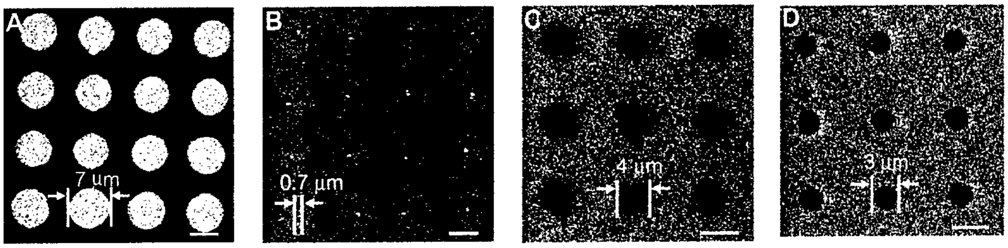

Figure 7. LFM images of (μCP-generated patterns before and after electrochemical whittling. (A) Original MHA dots array. (B) The MHA dot array in "A" after applying a potential of -920 mV vs Ag/AgCl for 30 sec. C) Original ODT dot array. D) The ODT dot array in "C" after applying a potential of -1100 mV vs Ag/AgCl for 5 min. The bar is 5 mm.

Fabrication of Metal Nanostructures from DPN Generated Templates. We have developed a novel method for fabricating solid-state metal nanostructures from DPN generated templates (Figure 8). This strategy relies on the careful choice of patterned and passivating molecules. For example, arrays of nanostructures comprised of dioctadecane diselenide (ODSe) are patterned on a gold substrate, and the bare gold surface is subsequently passivated using ODT. By applying a selectively desorbing potential the ODSe patterns are stripped, and a nanoscale bare gold template is generated. Subsequently, various metal salts can be electrochemically reduced and deposited on the exposed gold surface. We have demonstrated this approach by generating arrays of Ag nanostructures on a gold substrate (Figure 9). Interestingly, the resolution of nanostructures fabricated by using this approach is only limited by the size of DPN generated features.

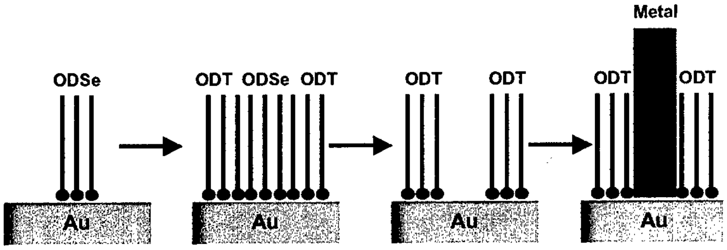

Figure 8. Alkylselenol patterns are selectively stripped, and the freshly exposed gold areas template the electrochemical growth of various metal salts.

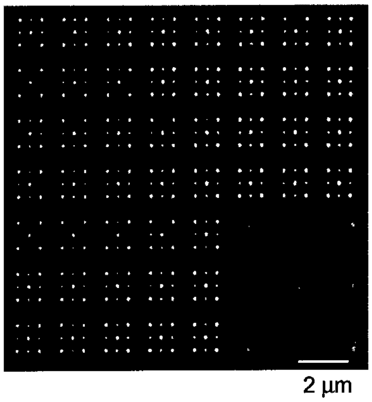

Figure 9. Darkfield optical micrograph of Ag nanostructures electrochemically grown on a thin film of Au. (Inset) TM-AFM image of the same nanostructures.

3. PERSONNEL SUPPORTED

Postdocs	Percentage of Salary Provided by Grant
None	0%

Graduate Students:	Percentage of Salary Provided by Grant
Martin Masar	100% (2000-2003)

Undergraduate Students:	Percentage of Salary Provided by Grant
None	0%

Unfunded Personel:	Percentage of Salary Provided by Grant
Khalid Salaita (grad)	100% (2003)
Yi Zhang (Post Doc)	100% (2003)

4. PUBLICATIONS

Peer-reviewed publications submitted and/or accepted during the 12-month period.

A. Journal Articles

1. Ivanisevic, A.; Im, J.H.; Lee, K-B.; Park, S.-J.; Demers, L.M.; Watson, K.J.; Mirkin, C.A. "Redox-Controlled Orthogonal Assembly of Charged Nanostructures" *J. Am.Chem.Soc.* **2001,** *123*, 12424-12425.
2. Zhang, Y.; Salaita, K.; Lim, J.-H.; Mirkin, C. A. "Electrochemical Whittling of Organic Nanostructures." *Nano Letters*, **2002**, *2*, 1389-1392.
3. Masar III, M. S.; Ovchinnikov, M.V.; Mirkin, C.A. "Fine-Tuning the "Weak-Link" Approach: Effect of Ligand Electron Density on the Formation of Rh^I and Ir^I Metallomacrocycles," *Inorg. Chem.* **2003**, *42*, 6851-6858.
5. Zhang, Y.; Salaita, K.; Lim, J.-H.; Lee, K.-B.; Mirkin, C. A. "A Massively Parallel Electrochemical Approach to the Miniaturization of Organic Micro- and Nanostructures on Surfaces." *Langmuir*, **2003**, submitted.

6. INTERACTIONS

A. Presentations/Presentations at Meetings, Conferences, Seminars

1. ACS National Meeting, Washington, D.C. "Functional Nanostructures," "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA" (2000)
2. Simon Fraser University, Vancouver, B.C.: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA" (2000)
3. NRL, Washington, D.C.: "Programming the Formation of Two-and Three-Dimen-sional Nanostructures with DNA" (2000)
4. University of Delaware, Biomaterials Conference, Newark, DE: Plenary Lecturer "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA" (2000)

5. NRL, Washington, D.C.: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA" (2000)
6. California Institute of Technology, Los Angeles, CA: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA" (2000)
7. MURI Review, Duck Key, FL: "Chemically Templated Colloidal Assembly via DPN" (2001)
8. Gordon Research Conference, Polymers West, Ventura, CA: "Dip-Pen Nanolithography" (2001)
9. 14th Biennial Marvel Symposium, University of Arizona, AZ: "Programmable Inorganic Architectures from DNA-Functionalized Building Blocks" (2001)
10. Pittcon 2001, New Orleans, LA: "Redox-Active and Inactive Nanostructures Generated via Dip-Pen Nanolithography" (2001)
11. NSF "Partnership in Nanotechnology" review, Arlington, VA: "DNA – directed Formation of Inorganic Nanostructures," "Non-Conventional Patterning Below 50nm" (2001)
12. AFOSR/ONR Electrochemistry Science & Technology Review, Annapolis, MD: "Nanostructured Materials for 3-D Structures" (2001)
13. University of Rochester, Rochester, NY: "Programming the Formation of 2- and 3-Dimensional Inorganic Architectures with DNA" (2001)
14. ACS, San Diego, CA: Symposium on Macromolecular Self-Assembly and Surfaces and Interfaces: "Dip-Pen Nanolithography: A new tool for studying template-driven particle assembly and crystallization" (2001)
15. 75th ACS Colloid and Surface Science Symposium, Pittsburgh, PA: "Dip-Pen Nanolithography: A Tool for Generating Organic and Biological Surface Architectures witch 5 nm Resolution" (2001)
16. DARPA review, Duck Key, FL: "Dip-Pen Nanolithography State-of-the-Art Applications and Future Challenges" (2001)
17. MURI review, Duck Key, FL: "Dip-Pen Nanolithography for Processing and Functionalizing Inorganic Semiconductor Substrates" (2001)
18. MRS, San Francisco, CA: "Terthienyl and Polyterthienyl Ligands as Redox Switchable Hemilabile Ligands for Oxidation-State Dependent Molecular Uptake and Release"; "Two- and Three- Dimensional DNA-driven Assembly of Colloidal Materials"; "Core-Shell Nanoparticles Formed from Ring- Opening Metathesis Polymerization and Functional Biomolecules"; "Dip-Pen Nanolithography and Combinatorial Nanotechnology" (2001)
19. ACS Colloid and Surface Science Symposium, U of Pittsburgh, PA: "Dip-Pen Nanolithography: A tool for generating organic and biological surface architectures with a 5nm resolution." (2001)
20. Gordon Research Conference, Polymers (East), New Hampshire: "2 and 3 D Nanoparticle Arrays" (2001)
21. Carnegie Melon University, Pittsburgh, PA: "Dip-Pen Nanolithography" (2001)
22. Kilpatrick Symposium on Nanotechnology, Chicago, IL: "Dip Pen Nanolithography: A Route towards Combinatorial Nanotechnology" (2001)
23. DARPA workshop, Charleston, SC: "Massively Parallel Dip-Pen Nanolithography" (2001)

24. NSF Sensor Workshop, Arlington, VA: "The New Challenges of Chemical and Biological Sensing" (2002)
25. DOE Workshop, San Diego, CA: "Biomolecular Materials" (2002)
26. Gordon Research Conference, Electrochemistry, Ventura, CA: "Dip-Pen Nanolithography Towards Combinatorial Nanotechnology" (2002)
27. 223rd ACS National Meeting, Orlando, FL: "Three dimensional assemblies formed via the Weak- Link Approach" (2002)
28. Particles 2002, Orlando, FL: "Ultrasensitive and Selective DNA and Protein-Based Detection by Nanoparticles" (2002)
28. Baekeland Awards Symposium and Presentation, Rutgers University, NJ: Keynote Speaker "Nanostructure-based Biodiagnostics" (2002)
29. NSF Workshop, Washington, D.C.: "Small Wonders: Exploring the Vast Potential of Nanoscience", "Small is Different...From Materials to Medicine" (2002)
30. DARPA/MTO Advanced Lithography Program Review, New Orleans, LA: "Dip-Pen Nanolithography" (2002)
31. International Symposium on Bioanalytical Chemistry and Nanotechnology, Hunan University, China: Keynote Speaker, "Nanoparticle Probes: A New Frontier in Biodetection" (2002)
32. Gordon Research Conference: Combinatorial Chemistry, Oxford, United Kingdom: "Dip-Pen Nanolithography and Combinatorial Nanotechnology" (2002)
33. Gordon Research Conference: Electronic Processes in Organic Mater, Salve Regina University, RI: "Dip-Pen Nanolithography and Combinatorial Nanotechnology" (2002)
34. University of Illinois, Urbana/Champagne CNST Lecture, Urbana, IL: "Massively Parallel Dip Pen Nanolithography: Towards Combinatorial Nanotechnology," (2002)
35. Washington University Chemistry Biology Interface Lecturer 2002, St. Louis, MO: "Nanoparticle-based Molecular Diagnostics: A New Frontier in Biodetection,"
36. NSF Workshop "Approaches to Combat Terrorism", Washington, D.C.: (2002)
37. MRS 2002 Fall Meeting, Boston, MA: "Scanning Multiplexed Raman Detection of DNA, RNA, and Protein Targets with Nanoparticle Probes"; "Biodirected Synthesis of Functional Materials using Nanoscale Building Blocks" (2002)
38. Nanoimprint and Nanoprint Technology Conference, San Francisco, CA: "Dip-Pen Nanolithography: Towards Combinatorial Nanotechnology" (2002)
39. DARPA Program Review - Biometric, Biomaterial, and Biointerfacial Sciences Program, Hawk's Cay, FL: "Ultra-Sensitive and Selective Chip-Based Detection of DNA"; "Surface Templated, Bio-Inspired Synthesis and Fabrication of Functional Materials" (2003)
40. PittCon Conference, Orlando, FL: "Building a High Tech Company in a University Environment"; "Novel Nanoparticle-Based Approaches to High Sensitivity Biomolecule Detection" (2003)
41. ACS National Meeting, New Orleans, LA: "Nanoparticle-Oligonucleotide conjugates: A new frontier in biodiagnostics" (2003)
42. Stanford University Student Hosted Colloquium Lecturer, Palo Alto, CA: "Massively Parallel Dip-Pen Nanolithography: Toward Combinatorial Nanotechnology" (2003)
43. DARPA Advanced Lithography Program Review, Santa Fe, NM: "Parallel, Ultrafast Sub-100 Nanometer Dip-Pen Nanolithography" (2003)

44. Frontiers in Chemistry – Mostafa El-Sayed Symposium, Atlanta, GA: "Light Assisted Routes to Anistropic Nanostructures" (2003)
45. ACS National Meeting, New York, NY: "Macrocycles Generated via the 'Weak-Link' Approach" (2003)

B. Consultative/Advisory Functions

1. Mirkin consults for and serves on the Scientific Advisory Board and the Board of Directors of both Nanosphere and NanoInk, Inc.

C. Transitions

1. NanoInk, Inc. has licensed the "electrochemical whittling" invention.

9. NEW DISCOVERIES, INVENTIONS, OR PATENT DISCLOSURES

Electrochemical whittling of lithographically defined structures. By Chad Mirkin, Yi Zhang, Khalid Salaita. New Invention Disclosure NU 22058. Disclosed on July 8, 2002.

10. HONORS/AWARDS

1. 2004 Nobel Laureate Signature Award for Graduate Education in Chemistry (2003)
2. Dickinson College Metzger-Conway Fellowship Award (2003)
3. 2003 Raymond and Beverly Sackler Prize in the Physical Sciences (NU, 2003)
4. Forbes.com, The Forbes/Wolfe Nanotech Report, Mar 2003 "Nanotechnology's Top 10 Power Brokers"
5. 2002 Feynman Prize in Nanotechnology (NU, 2002)
6. 2002 Ceramographic Competition, American Ceramic Society 1st Place Entry (NU, 2002)
7. Esquire Magazine's "Best & Brightest in the Nation" (NU, 2002)
8. 2001 Leo Hendrick Baekeland Award (NU, 2001)
9. Crain's Chicago Business "40 under 40 Award" (NU, 2001)
10. Discover 2000 Award for Technological Innovation (NU, 2000)
11. Elected Fellow of the American Association for the Advancement of Science (NU, 2000)
12. I-Street Magazine's Top 5 List for Leading Academics in Technology (NU, 2000)