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"Nanostructured Materials for 3-D Powerstructures"

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13. ABSTRACT *(Maximum 200 words)*

We have been able to make the necessary compounds and nanoparticles in the synthetic portion of the proposal. We were able, for the first time, to successfully link DNA to organic polymers and study their interaction. Further, we have been able to manipulate the resulting structures to incorporate gold nanoparticles. We are in the process of applying these building blocks to build up a 3-D structure using DNA-recognition and DPN-based patterning.

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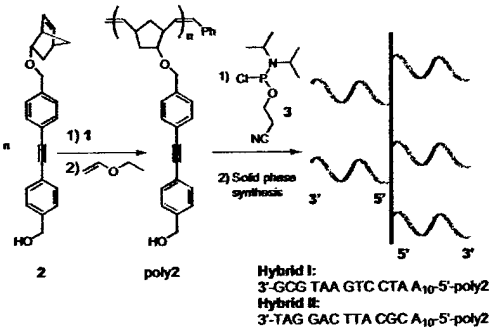
1. Objectives of Research. 1) To develop the fundamental surface science, materials, and electrochemistry necessary for synthesizing nanoscale inorganic/organic composite components for 3-D power structures, 2) to extend DNA-based assembly strategies to these novel nanocomponent materials, 3) to characterize and evaluate the electrochemical and general physical properties of the 3-D power structures prepared via the novel assembly strategies described herein, and 4) to develop and utilize dip-pen nanolithography as a powerful tool that will help in the realization of objectives 1-3.

2. Status of Effort. We have been able to make the necessary compounds and nanoparticles in the synthetic portion of the proposal. We were able, for the first time, to successfully link DNA to organic polymers and study their interaction. Further, we have been able to manipulate the resulting structures to incorporate gold nanoparticles. We are in the process of applying these building blocks to build up a 3-D structure using DNA-recognition and DPN-based patterning.

3. Accomplishments.

1. New DNA-functionalized ROMP polymers have been prepared by the post-polymerization modification of an organic polymer containing DNA-linkers developed in our laboratory (ROMP = ring-opening metathesis polymerization). In this methodology, the diphenylacetylenebenzyl alcohol-substituted norbornene **2** was first polymerized using catalyst **1**, $Cl_2Ru(PCy_3)_2CHPh$. Termination (by the injection of ethyl vinyl ether) and post-polymerization modification of the resulting polymer with a chlorophosphoramidite **3** following by solid phase DNA synthesis led to the isolation of hybrid materials such as **Hybrid-I** and **Hybrid-II** (Scheme 1). These materials exhibit excellent DNA recognition properties when combined with each other and when combined with nanoparticles containing complementary DNA strands.

Scheme 1



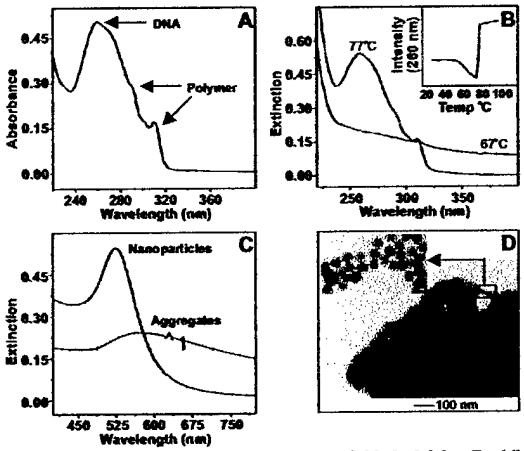
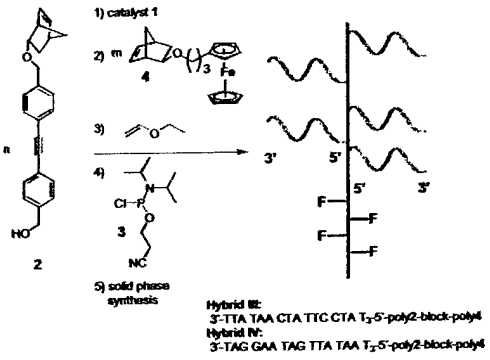


Figure 1. A. UV-Vis spectrum of **Hybrid-I**. B. UV-Vis spectra before and after melting of **Hybrid-I/Hybrid-II** mixture (melting curve inset) C. UV-Vis spectra of DNA-modified 13-nm Au nanoparticles and aggregates of **Hybrid-I** and complementary DNA-modified 13-nm Au nanoparticles. D. TEM image of the aggregates from C.

2. Block copolymers containing DNA also can be synthesized using a protocol similar to the one described above but with a subsequent addition (and polymerization) of a second norbornene monomer, such as the ferrocenyl-containing monomer **4**, prior to the

termination step. This yields block copolymers with the desired di-block structure. Reaction with the chlorophosphoramidite **3** followed by solid phase DNA synthesis led to the isolation of block copolymer hybrid materials such as **Hybrid-III** and **Hybrid-IV** (Scheme 2). As depicted in Figure 2, the electrochemistry and hybridization behavior of these hybrids are quite well-defined.

Scheme 2



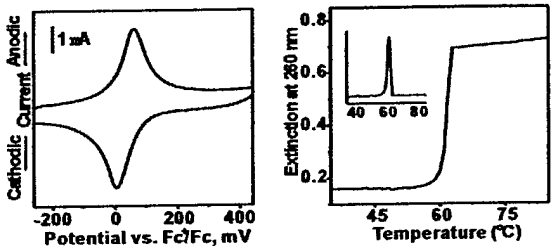


Figure 2. (A) The cyclic voltammogram of **Hybrid IV** in CH_2Cl_2. (B) The melting curve for **Hybrid-III/Hybrid-IV** in a PBS buffer (first derivative inset).

3. An unanticipated offshoot of this work was the development of a new DNA detection system based on these novel materials. For example, Hybrids **III** and **IV** can be used as electrochemical probes for the detection of DNA. Figure 3 illustrates our ability to detect DNA at nM concentration using polymer probes, and points to a new route for biowarfare detection.

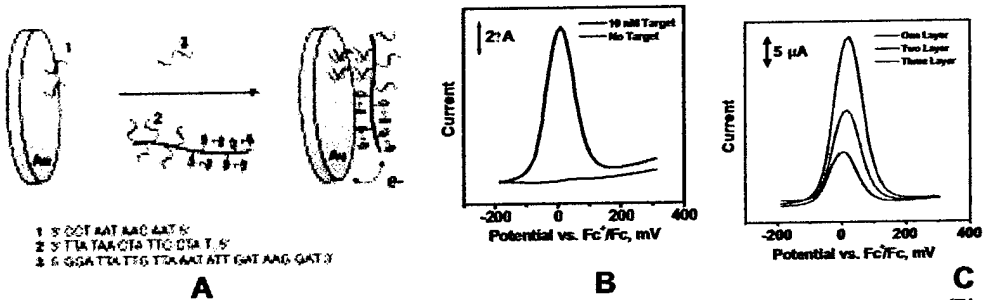


Figure 3. (A) A DNA detection scheme with ferrocenyl-substituted polymer probe. (B) Electrochemical detection of nM concentration of DNA with one layer of polymer. (C) Amplification of Electrochemical signal with additional layers of polymer.

4. The synthesis of other ferrocene derivatives has been heavily investigated in our laboratory with the intent to combine several substituted ferrocenes into DNA/polymer hybrid and use these to encode electrochemical properties into the resulting hybrid materials for potential signaling and diagnostic applications. The aim is to first develop a triblock polymer with a DNA block and two blocks of different ferrocene monomers with redox potentials approximately 200-350 mV apart. The difference in potentials would result in two distinct peaks in a cyclic voltammetry experiment.

This technology can then be extended to individually label each nanoparticle with combinations of ferrocene "flavors" much akin to "bar-coding" the particles: the position and magnitude of the electrochemical peaks associated with each particle would be proportional to the types of monomer present in the particle and the number of repeating units of that particular monomer. Each of these "bar-coded" polymer-coated particles

can then be associated with a specific sequence of DNA via parallel synthesis. Thus, by varying the ferrocene monomer composition and ratios in the multiblock polymers that coat the nanoparticles, we can create many distinct electrochemical labels for the detection of multiple DNA targets in an array fashion using the DPN-templated array described below.

5. The development of methods for organizing nanostructures into functional materials with addressable nanoscopic components represents a significant challenge in nanoscience. A variety of methods have been employed to control the assembly of nanoparticles into ordered two- and three-dimensional architectures in solution and on surfaces. However, at present there are no efficient methods for chemically directing the assembly of *multicomponent* nanostructures on surfaces with precision control over the placement of the nanoscale building blocks. An intriguing possibility for the bio-molecule based approach to particle assembly is that if one could learn how to pattern biological molecules on surfaces with nanoscale resolution, one literally could chemically program or encode such surfaces with information based upon the biorecognition elements used in the patterning process. For example, in the case of a synthetic sequence of DNA that is 20 bases long, there are 4^{20} possible recognition elements that could be used for guiding the assembly of nanoscale building blocks functionalized with the appropriate complementary sequences. As the length of the sequence increases, the number of recognition elements dramatically increases providing almost a limitless number of interaction pairs that can be designed to guide a given nanostructure assembly process. In contrast, if one were to use ordinary organic molecules and either surface modification chemistry or covalent organic methods for directing such processes, there are a limited and small number of interaction pairs that could be designed and employed. Our efforts this year were directed towards using DPN to create nanostructures of DNA on a surface, which can be subsequently used to guide the assembly of discrete nanoparticle building blocks with complementary DNA in an orthogonal manner. Significantly, this strategy, once perfected, could lead to a new and general way for preparing multicomponent nanostructures for a wide range of applications ranging from biological diagnostics to nanoelectronics to the preparation of colloidal crystals for use in catalysis, power structures and photonics.

   Our general synthetic strategy is outlined in Figure 4. DPN can be used as a tool for generating combinatorial chemical templates with which to position single particles in two-dimensional arrays. It is important to note that the DPN approach will allow one to systematically vary array chemical composition, spot size, spot shape, and inter-feature distances. For example, charged alkanethiols and latex particles have been used in a proof-of-concept experiment, in which we have demonstrated that this general approach can be used to create useful two-dimensional templates for positioning subsequent particle layers in predefined crystalline structures that may be composed of single or multiple particle sizes and compositions (Figure 5).

   In a more general sense, the combinatorial DPN method allowed us to efficiently and quickly form patterned substrates with which to study particle-particle, and particle-substrate interactions, whether the particles are the dielectric spheres which comprise certain photonic band-gap materials metal or semiconductor particles with potential catalytic or electronic properties. If two different oligonucleotides are used on the gold,

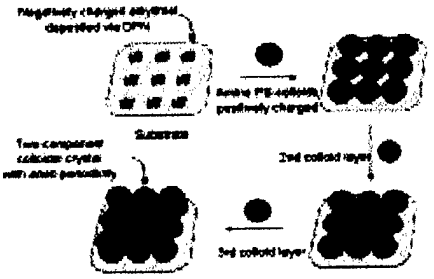


Figure 4. A general strategy for organizing building blocks into three-dimensional arrays.

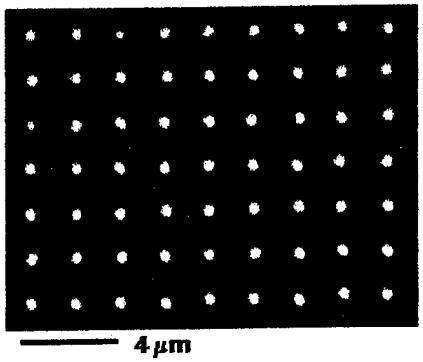


Figure 5. Polystyrene particles assembled on to a DPN pattern via electrostatic interaction

two different sizes of spheres, functionalized with the complementary oligonucleotides can be arranged in organized arrays onto the substrate (Figure 6). We have demonstrated this initial concept by using two different oligonucleotide-functionalized gold nanoparticles (Figure 7). Initial experiments show that the assembly process is almost perfect! Subsequent layers of any composition can be added by using particles that are functionalized with DNA that is complementary to the first layer. Repeating this procedure will lead to a multilayer structure. The self-assembly process, applied in this fashion, should lead to a variety of interesting colloidal crystals, including simple cubic, NaCl and CsCl structure, as well as AB_2 structures. Again, the key to this strategy is that oligonucleotide-oligonucleotide interactions are strong enough to surface immobilize particles, but weak enough to allow particle equilibration and migration on the surface to form the predetermined thermodynamic structure. Importantly, the speed of the equilibration process can be controlled by temperature, solvent, and choice of DNA sequence. We are now beginning to define the electron transport properties of these structures.

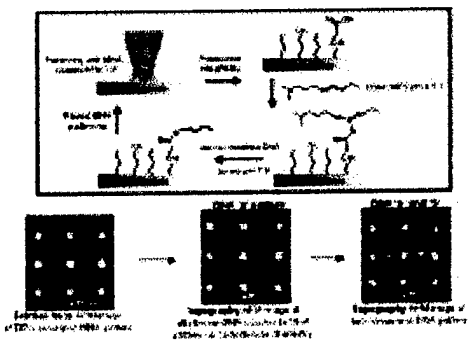


Figure 6. The process behind the functionalization of a surface with DNA. The lower images show the patterned surface at each step during the process.

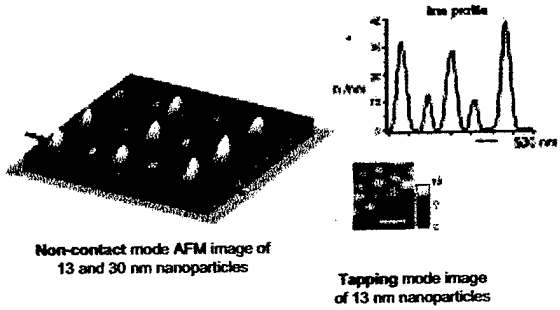


Figure 7. Orthogonal assembly of 13- and 30-nm DNA-functionalized Au particles.

6. Key to this research effort is an understanding of the electronic and electrical properties of DNA-directed assembly hybrid which can potentially be used in the constructions of 3-D power structures. A primary question is: can structures assembled by DNA-directed

nanoparticles assembly conduct electricity? To this end, we have found that the use of DNA to assemble nanoparticle-based materials do not passivate the structural and electrical properties of the nanoparticles. Further, aggregates with the DNA-protected nanoparticles tend to behave as semiconductors, with conductivities varied predictably as functions of moisture and salt concentration. Finally, we have shown that the conductivities of DNA-Au nanoparticle assemblies are virtually independent of the length of the linker when solvent and salt are removed and allows the DNA to be compressed on the surface, bringing the particles closer within the aggregates. These results point toward the intriguing possibility of using these materials in power applications where changes in interparticle environment can lead to dramatic increases in conductivity (i.e. from a conductor to an insulator or a semiconductor).

4. Personnel Supported by Grant:

Principal Investigators:	Chad A. Mirkin
	SonBinh T. Nguyen
Postdocs:	Seunghun Hong
Graduate Students:	Keith J. Watson
	So-Jung Park
	Julianne M. Gibbs

5. Publications (10/1/00-present):

1. *"The Synthesis and Ring-Opening Metathesis Polymerization of an Amphiphilic Redox-Active Norbornene"* Watson, K. J.; Nguyen, S. T.; and Mirkin, C. A. *J. Organomet. Chem.* **2000**, *606*, 79-83.
2. *"DNA-Block Copolymer Conjugates"* Watson, K. J.; Park, S.-J.; Ihm, J.-H.; Nguyen, S. T.; and Mirkin, C. A. *J. Am. Chem. Soc.*, **2001**, *123*, 5592-5593.
3. *"Function Targeted Monomer Development for Ring-Opening Metathesis Polymerization: Fundamentals and Applications"* Watson, K. J. Ph. D. Thesis, Northwestern University, May 2001.
4. *"Toward Polymeric Anti-Cancer Drug Cocktails from Ring-Opening Metathesis Polymerization"* Watson, K. J.; Anderson, D. R.; and Nguyen, S. T.; *Macromolecules*, **2001**, *34*, 3507-3509.
5. *"Combinatorial Generation and Analysis of Nano- and Micron Scale Silicon Features Via "Dip-Pen" Nanolithography and Wet Chemical Etching"* Weinberger, D. A.; Hong, S.-H.; Mirkin, C. A.; Wessels, B. W.; Higgins, T. B. *Adv. Mater.* **2000**, *12*, 1600-1603.
6. *"Directed assembly of periodic materials from protein and oligonucleotide-modified nanoparticle building blocks"* Park, S.-J.; Lazarides, A. A.; Mirkin, C. A.; Letsinger, R. L. *Angew. Chem., Int. Ed. Engl.* **2001**, *40(15)*, 2909-2912.
7. *"The electrical properties of gold nanoparticle assemblies linked by DNA"* Park, S.-J.; Lazarides, A. A.; Mirkin, C. A.; Brazis, P. W.; Kannewurf, C. R.; Letsinger, R. L. *Angew. Chem., Int. Ed. Engl.* **2000**, *39(21)*, 3845-3848.
8. *"Haplotyping by Force"* Taton, T. A.; Mirkin, C. A. *Nat. Biotech.* **2000**, *18*, 713.
9. *"Dip-pen Nanolithography: Controlling Surface Architecture on the Sub-100 Nanometer Length Scale"* Mirkin, C. A.; Hong, S.; Demers, L. M. *Chem. Phys. Chem.* **2001**, *2*, 37-39.

10. "*Combinatorial Templates Generated by Dip Pen Nanolithography for the Formation of Two-Dimensional Particle Arrays*" Demers, L. M.; Mirkin, C. A. *Angew. Chem., Int. Ed. Engl.* **2001**, 40(16), 3069-3071.
11. "*Orthogonal Assembly of Nanoparticle Building Blocks on Dip-Pen Nanolithographically Generated Templates of DNA*" Demers, L. M.; Park, S.-J.; Taton, T. A.; Li, Z.; Mirkin, C. A. *Angew. Chem., Int. Ed. Engl.* **2001**, 40(16), 3071-3073.

6. Interactions/Presentations

Chad A. Mirkin

1. Georgia Institute of Technology, Atlanta Georgia, ONR Program Review: "The Weak Link Approach to the Synthesis of Homobimetallic Macrocycles", 2000.
2. MURI (ACM) Project, Program Review Meeting, Northwestern University, Evanston, IL: "Biosensors and Bioinorganic Hybrid Materials", 2000,.
3. MURI 2K Kickoff Meeting, Northwestern University "Surface Templated, Bio-Inspired Synthesis and Fabrication of Functional Materials", 2000.
4. University of Essen, DFG-Workshop on Nanostructured Surfaces, Aachen, Germany "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
5. DuPont, Wilmington, DE: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
6. DARPA MEMS, Highlands Forum XIV, Washington, DC "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
7. NIH, BECON Symposium, Application of Nanotechnology" Bethesda, MD: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
8. CMACS2000, Symposium on New Materials for Electronics, Cincinnati, OH: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
9. NSF Molecular Electronics Workshop, Arlington, VA: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
10. DARPA, FOCUS 2000, Chantilly, VA: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
11. Gordon Research Conference, Bioanalytical Sensors, Proctor Academy, Andover, NH: "Ultraselective and Sensitive Nanoparticle-Based DNA Detection Methods", 2000.
12. Gordon Research Conference, Organic Structures & Properties, Connecticut College, New London, Connecticut: Discussion Leader, 2000.
13. ACS, Washington, DC "Functional Nanostructures": "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
14. University of British Columbia, Vancouver, BC: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
15. Simon Fraser University, Vancouver, BC: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
16. University of Delaware, Biomaterials Conference, Newark, DE: Plenary Lecturer "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.

17. NRL, Washington, DC: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
18. CalTech, LA, CA: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2000.
19. MRS, Boston, MA: "Nanoparticle-Based Detection Methods for DNA", 2000,.
20. MRS, Boston, MA: "Parallel Dip-Pen Nanolithography: A New Tool for Generating and Studying Soft Nanostructures", 2000.
21. Pacifichem 2000, Hawaii: "The "weak link" approach to designing 2-D and 3-D cage structures with tailorable molecular recognition properties", 2000.
22. Gordon Research Conference, Polymers West, Ventura, CA: "Dip-Pen Nanolithography", 2001.
23. 14th Biennial Marvel Symposium, University of Arizona, AZ: " Programmable Inorganic Architectures from DNA-Functionalized Building Blocks", 2001,.
24. Pittcon 2001, New Orleans, LA: "Redox-Active and Inactive Nanostructures Generated Via Dip-Pen Nanolithography", 2001.
25. NSF "Partnership in Nanotechnology" review, Arlington, VA: "DNA directed Formation of Inorganic Nanostructures, 2001
26. NSF workshop: Non-Conventional Patterning Below 50nm, Arlington, VA, 2001.
27. AFOSR/ONR Electrochemistry Science & Technology Review, Annapolis, MD: "Nanostructured Materials for 3-D Structures", 2001,.
28. University of East Carolina, Greenville, NC: "Programming the Formation of Two-and Three-Dimensional Nanostructures with DNA", 2001.
29. University of Rochester, Rochester, NY: "Programming the Formation of 2- and 3-Dimensional Inorganic Architectures with DNA", 2001.
30. Electrochemical Society Meeting, Washington, DC: "Scanometric DNA Array Detection with Nanoparticle Probes", 2001.
31. ACS San Diego Symposium on Macromolecular Self-Assembly and Surfaces and Interfaces, San Diego, CA: "Dip-Pen nanolithography: A new tool for studying template-driven particle assembly and crystallization", 2001.
32. Gettysburg College, Gettysburg, PA: "Supramolecular Nanoscale Objects Prepared Via Coordination Chemistry; Nanotechnology: Thinking Small or Small Thinking; Nanostructures in DNA Detection", 2001.
33. 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.
34. DARPA review, Duck Key, FL: "Dip-Pen Nanolithography State-of-the-Art Applications and Future Challenges", 2001.
35. MURI review, Duck Key, FL: "Dip-Pen Nanolithography for Processing and Functionalizing Inorganic Semiconductor Substrates", 2001.
36. DARPA workshop, Charleston, SC: "Massively Parallel Dip-Pen Nanolithography", 2001.
37. Gordon Research Conference, Chemical Sensors & Interfacial Design, Lucca, Italy: "Nanoparticle-based probes: New opportunities in DNA diagnostics, gene expression, and arrays", 2001.
38. 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.

SonBinh T. Nguyen

1. "Metal Organic Hybrid Nanoparticle Structures" at the 2000 NSF Materials Chemistry Workshop (October 12-15, 2000), Mount Hood, OR.
2. "The Catalytic Asymmetric Coupling of Monofunctional Groups and C_2 Molecules" October 18, 2000 Seminar at California Institute of Technology, Pasadena, CA.
3. "The Catalytic Asymmetric Coupling of Monofunctional Groups and C_2 Molecules" October 25, 2000 Seminar at Stanford University, Palo Alto, CA.
4. "The Catalytic Asymmetric Coupling of Monofunctional Groups and C_2 Molecules" October 25, 2000 Seminar at Stanford University, Stanford, CA.
5. "Metal Organic Hybrid Nanoparticle Structures: Synthesis and Applications" November 09, 2000 Colloquium at Ball State University, IN.
6. "Catalytic Asymmetric Synthesis of Cyclopropanes and Epoxides from Olefins" November 15, 2000 seminar at Emory University, Atlanta GA.
7. "Catalytic Asymmetric Synthesis of Cyclopropanes and Epoxides from Olefins" November 16, 2000 seminar at Georgia Institute of Technology, Atlanta GA.
8. "Functionalized Nanoporous Membranes" at the 2001 NSF Partnership in Nanotechnology Conerence (January 29-30, 2001), Arlington, VA.
9. "The Catalytic Asymmetric coupling of Monofunctional Groups and C_2 Molecules" February 20, 2001 seminar at University of California, Berkeley CA.
10. "Olefin Cyclopropanation and Artificial Enzymes for Olefin Epoxidation" April 12, 2001 seminar at Pharmacia Corporation, Skokie, IL.
11. "The Polymer Module in the Materials World Module Series: A Design-Oriented Approach to Incorporating Polymer Chemistry in the General Science Curriculum of Grades 9-12" at the 2001 MRS Spring Meeting (April 16-20, 2000), San Francisco, CA.
12. "Asymmetric Olefin Cyclopropanation Catalyzed by Group VIII Metal Complexes" at the 221st ACS National Meeting (April 1-5, 2001), San Francisco, CA.
13. "Supramolecular Effects in Olefin Epoxidation Chemistry: Towards the Design of an Artificial Enzyme" at the 221st ACS National Meeting (April 1-5, 2001), San Francisco, CA.
14. "Teaching Polymer Science" at the NSF-NUMRC Teaching Concepts Through Materials Conference (June 4-5, 2001), Evanston, IL
15. "Transfer Hydrogenation of Ketones and Aldehydes using Simple Aluminum Catalysts" at the 222nd ACS National Meeting (August 26-30, 2001), Chicago, IL.
16. "Multidentate Ligands in Supramolecular Chemistry" at the 222nd ACS National Meeting (August 26-30, 2001), Chicago, IL.

7A. Consulting

1. Consultation with Shashi Karna (AFRL) about application of polymer in nanotechnology. (Nguyen, Mirkin)
2. Founder and Chair of Scientific Advisory Board Nanosphere, Inc. (Mirkin)
3. Founder and Chair of Scientific Advisory Board, NanoInk, Inc. (Mirkin)

4. Editor Selection Committee, Journal American Chemical Society (Mirkin)
5. Committee Member, NSF Workshop on New Frontier in Inorganic Chemistry
6. Consultation with Paul Sheehan (NRL) regarding DPN.

7B. Transition

NRL (Colton, Sheehan) and AFRL (Morley Stone) are now using DPN, based on consultation with Mirkin.

8. Patents

1. "*DNA-Block Copolymer Conjugates*" Mirkin, C. A.; Nguyen, S. T.; Watson, K. J.; Park, S.-J. , Filed 2000.

9. Honors and Awards (1/1/00-present)

Chad A. Mirkin

2000: Appointed George B. Rathmann Professor of Chemistry
2000: Appointed Director of the Institute for Nanotechnology
2000: Discover 2000 Award for Technological Innovation
2000: Elected Fellow of the American Association for the Advancement of Science
2000: I-Street Magazine's Top 5 List for Leading Academics in Technology

SonBinh T. Nguyen

2001: Alfred P. Sloan Research Fellow
2000: Presidential Early Career Award for Scientists and Engineers (from AFOSR)
2000: National Science Foundation CAREER Award