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14. ABSTRACT This proposal was directed toward studying the electrical and thermal transport in carbon nanotube devices and array assemblies with the ultimate goal of investigating the possibility of a measurable Peltier effect in carbon nanotube devices. This final report will describe the effort performed during the period of this project. The single walled carbon nanotubes for this project were grown in-house by chemical vapor deposition (CVD).					
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Report Title

Final Report: Novel Nanoscale Materials for Energy Conversion Applications

ABSTRACT

This proposal was directed toward studying the electrical and thermal transport in carbon nanotube devices and array assemblies with the ultimate goal of investigating the possibility of a measurable Peltier effect in carbon nanotube devices. This final report will describe the effort performed during the period of this project.

The single walled carbon nanotubes for this project were grown in-house by chemical vapor deposition (CVD). CVD synthesis has many advantages over other methods for the fabrication of nanotube devices. CVD-grown nanotubes can be much longer and cleaner than bulk-grown nanotubes that are deposited from solution, and can be grown from patterned catalyst islands. It is possible to control the nanotube diameter by controlling the nanoparticle size, and the placement of CVD grown CNTs can be controlled by patterning the catalysts. Control of CNT properties is potentially possible but challenging as the understanding of the process or the mechanism that would control the chirality of individual tubes is not complete.

The project involved development of a CVD synthesis facility to grow the nanotubes, patterning and fabrication of nanotube devices using dip pen nanolithography (DPN) and electron beam lithography, and characterization of the resulting nanotubes and devices by a variety of techniques.

List of papers submitted or published that acknowledge ARO support during this reporting period. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Kuljanishvili, I. Dikin D., S. Rozhok, S. Mayle, V. Chandrasekhar,; Controllable Patterning and CVD Growth of Isolated Carbon Nanotubes with Direct Parallel Writing of Catalyst Using Dip-Pen Nanolithography, Small, 5, 22, 2523-2527. 2009, Cover article.

Number of Papers published in peer-reviewed journals: 1.00

(b) Papers published in non-peer-reviewed journals or in conference proceedings (N/A for none)

Number of Papers published in non peer-reviewed journals: 0.00

(c) Presentations

1) "Synthesis and characterization of SWCNT prepared on silicon substrate with different methods of patterning catalyst particles", I. Kuljanishvili, O. Loh, D. Dikin, H. Espinosa, R. Piner, R. S. Ruoff and V. Chandrasekhar, March Meeting of the Am. Phys. Soc., New Orleans, Louisiana, 2008 (oral presentation).

2) "CVD grown of CNTs on Si substrate from DPN patterned catalyst precursor", I. Kuljanishvili, D. Dikin, S. Rozhok, S. Mayle and V. Chandrasekhar, March Meeting of the Am. Phys. Soc., Pittsburg, Pennsylvania, 2009 (oral presentation).

3) "Controllable Patterning and CVD Growth of Carbon Nanotubes with Direct Parallel Writing of Catalyst Ink using Dip-Pen Nanolithography", I. Kuljanishvili, D. Dikin, S. Rozhok, S. Mayle and V. Chandrasekhar, MRS Spring Meeting, San Francisco, California, 2010 (oral presentation)

Number of Presentations: 3.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts): 0

Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Number of Manuscripts:0.00

Patents Submitted

Patents Awarded

Awards

Venkat Chandrasekhar, Fellow of the American Physical Society

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
Scott Mayle	0.00
FTE Equivalent:	0.00
Total Number:	1

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
Irma Kuljanishvili	1.00
FTE Equivalent:	1.00
Total Number:	1

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	National Academy Member
Venkat Chandrasekhar	0.02	No
FTE Equivalent:	0.02	
Total Number:	1	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
Pavan Patel	0.00
Alice Rice	0.00
Jesse Choe	0.00
Rachel Koltun	0.00
Dima Spivak	0.00
Alex Carter	0.00
FTE Equivalent:	0.00
Total Number:	6

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 3.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 3.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 2.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale): 2.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 2.00

Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PhDs

NAME

Total Number:

Names of other research staff

NAME

PERCENT SUPPORTED

FTE Equivalent:

Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Technology Transfer

Final Report for ARO proposal (August 2010)

Introduction

This proposal was directed toward studying the electrical and thermal transport in carbon nanotube devices and array assemblies with the ultimate goal of investigating the possibility of a measurable Peltier effect in carbon nanotube devices. This final report will describe the effort performed during the period of this project.

The single walled carbon nanotubes for this project were grown in-house by chemical vapor deposition (CVD). CVD synthesis has many advantages over other methods for the fabrication of nanotube devices. CVD-grown nanotubes can be much longer and cleaner than bulk-grown nanotubes that are deposited from solution, and can be grown from patterned catalyst islands. It is possible to control the nanotube diameter by controlling the nanoparticle size, and the placement of CVD grown CNTs can be controlled by patterning the catalysts. Control of CNT properties is potentially possible but challenging as the understanding of the process or the mechanism that would control the chirality of individual tubes is not complete.

The project involved development of a CVD synthesis facility to grow the nanotubes, patterning and fabrication of nanotube devices using dip pen nanolithography (DPN) and electron beam lithography, and characterization of the resulting nanotubes and devices by a variety of techniques.

Equipment built and purchased

The equipment built or purchased during this project included a home-built chemical vapor deposition system and a high resolution Scanning Probe Microscope obtained with DURIP funds.

Chemical vapor Deposition System (home-built)

The main components include of this system include:

- 1) Lindberg Blue Mini-Mite Tube furnace with a 1 inch quartz tube and compression fittings.
- 2) Sierra Instruments Model 954 Digital Flow Box power supply/ controller (calibrated to medium to high flow rate: 2400 sccm max) that can control up to 4 mass flow controllers.
- 3) Three mass flow controllers, Model 840L series, Sierra Instruments, Inc.
- 4) Gas handling system for argon, helium, hydrogen and methane.

The CVD system is used primarily for CNT production. It has also been used for various high temperature annealing processes, and most recently has been employed for CVD synthesis of graphene on nickel films.

Park Systems XE-150 Multimode SPM

Through a DURIP grant, a Park Systems XE-150 Multimode Scanning Probe Microscope was purchased and installed in July 2008. This system is designed for large size samples, with completely decoupled XY & Z scanners which use a flexure-guided scan system for all three axes, a closed/open loop scan, a zero background curvature XY flexure scanner, a XE AFM scanning head, direct on-axis optics, motorized Z focus stage, motorized long-range XY sample stage, acoustic enclosure, active vibration isolation, electronics controller, software and cantilevers. This machine is capable of performing AFM, MFM and EFM (among other modes), and has enabled us to image and locate single wall carbon nanotubes with diameters on the order of 1 nm, and graphene films with thicknesses less than 0.5 nm.

Other facilities employed

Raman Spectroscopy Instrumentation

The use of an InVia Raman spectroscope was requested under the user program at the Center of Nanoscale Materials (CNM) at Argonne National Laboratory for characterizing carbon nanotubes and graphene films. During the project time period a total of two user proposals were submitted:

1. CNM 856, entitled “Raman Spectroscopy Study of individual Single-walled Carbon Nanotubes prepared on silicon substrates.”
2. CNM 20402 entitled “Raman Spectroscopy Study of suspended Single wall Carbon Nanotubes.”

These studies were used to determine the properties of the single-walled carbon nanotubes.

Other instrumentation used

a) **Conventional patterning** (e-beam lithography.) The PI’s lab is equipped with a clean room with thermal and e-gun evaporators, and a Tescan field emission SEM, also purchased with another DURIP grant, that has been adapted for electron beam lithography

b) **Novel SPM based lithography techniques.**

The Dip Pen Nanolithography (DPN) platform NLP 2000 from NanoInk, Inc., was used for patterning the catalyst for CNT growth. This system employs multipen cantilevers for parallel writing capabilities. Access to the instrument was provided through a collaboration with NanoInk Inc.

Results and Analyses

Fabrication and Experimental methods

The cost of fabricating carbon nanotubes, dispersion in the nanotube type, and in particular limitations in processing and assembly methods are the key barriers for successful and reproducible production of single-walled nanotube devices and multi tube assemblies. During the course of this project we encountered and tried to surmount the key challenges mentioned above. In any study that involves mesoscopic and nanoscale devices, reproducibility and control over the fabrication process is essential. One of the major problems in fabricating devices from carbon nanotubes is due to their small size, which makes controlling the position of carbon nanotubes while preserving their quality very difficult.

We developed a protocol that employs photo- and e-beam lithography in combination with state-of-the-art technologies such as scanning probe based (SPM) patterning techniques, chemical vapor deposition (CVD) catalysis and conventional silicon processing methods. Fabrication of any true nanoscale device is challenging but more so when organic macromolecules such as carbon nanotubes are involved. The main task for production of CNT-based devices is the ability to produce CNTs of the desired length, diameter, and electronic properties at specific predefined locations. The CVD method of synthesis is the most convenient tool to be used for carbon nanotubes grown at specific chosen locations. One of the most critical points in fabrication of nanoscale devices is the development of the technology that can be flexible and scalable. Thus existing manufacturing technologies need to be incorporated with new methods and metrologies for more efficient and economic ways of device production.

The protocol which we use here can be divided into three main subsections: 1) Patterning of the layout and deposition of the catalyst at predefined locations; 2) CVD synthesis and characterization of carbon nanotubes and 3) device fabrication and testing.

Patterning of the layout and deposition of the catalyst with the Dip Pen Nanolithography (DPN) technique. The first stage of the fabrication process includes defining a grid of fiducial markers such as letters that identify specific locations on the substrate. This is typically accomplished by using conventional lithographic tools such as photo or e-beam lithography and standard lift off techniques. After this first fabrication step, the substrates undergo extensive cleaning and heat treatment in a Ar/H_2 environment at 450-500 C in order to remove residues of the e-beam resist or other organic contaminants. High melting point metals such as Cr or Ni are usually used to define these fiducial markers. In the next step we employ DPN to deposit catalyst at specifically chosen locations near the markers. The DPN technique we employed can be described as follows: the tip of an atomic force microscope (AFM) is dipped in an “ink” that can subsequently be transferred to a substrate with the nanometer-scale precision typical for scanning probe microscopes. This is

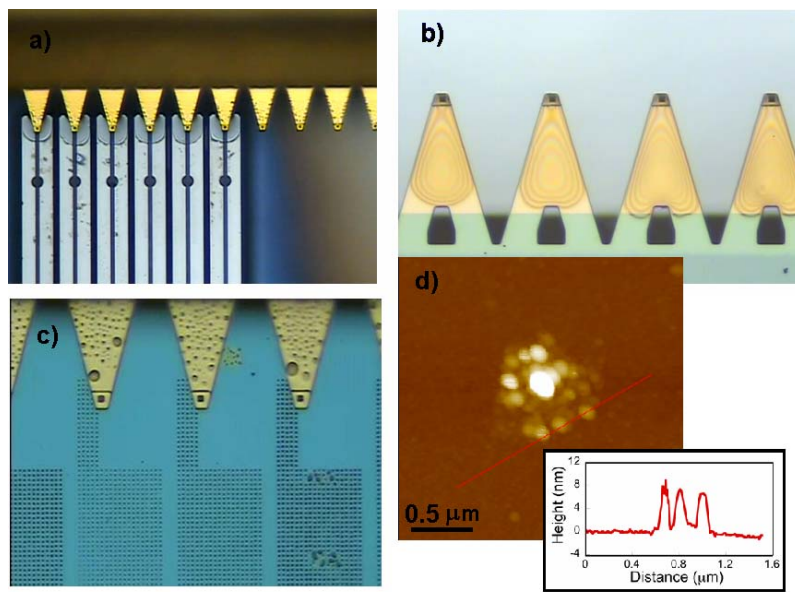


Fig. 1. Optical images of a) 6 tips dipped in ink wells for inking at one time, b) bottom view of 4 tips on the cantilever, c) Simultaneous patterning by a 12 pen cantilever array (only 3 pens are shown on the picture). Distance between cantilevers tips is $\sim 60 \mu\text{m}$, spacing between dots in each array for was $5 \mu\text{m}$. d) AFM topographic image of individual DPN catalyst dot cluster after annealing. Inset shows topographic line profile across the red line for three Fe nanoparticles, indicating the typical size of $\sim 6\text{-}8 \text{ nm}$.

a mask free technology that allows for convenient high precision patterning. An iron-based molecular “ink” that is delivered by a parallel “writing” method employing multi-pen cantilevers was developed in our earlier research. This “ink” is optimized to produce high-quality isolated CNTs for easy integration into various device architectures. The ink was prepared by mixing a water-based master solution of ferric nitrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ with dimethylformamide (DMF) and glycerol to promote long writing times and minimize the evaporation of the ink. Fig. 1 shows the process of “inking and printing” of arrays of dots. In this manner

arrays of CNT structures can be produced. With a single dip into the inkwell (that is a separate microfabricated structure made of silicon that holds ink solution reservoirs) an array of approximately 10^4 dots can be written by a single cantilever tip. For producing an array of dots over a large area of the substrate, the cantilever tips can be repeatedly dipped into the inkwells, allowing automated patterning of large arrays over a span of hours. Fig. 1 a) and b) shows the inking process. The patterns are designed in a software program and can therefore be changed readily, and large arrays of identical structures can be fabricated by simultaneously using multiple tips in an ordered array, as seen in Fig. 1 c). Patterning by an individual tip-pen is quite uniform. However, occasional variations in writing performed by a particular pen are observed, as seen in Fig. 1c.

CVD Synthesis of Single Wall Carbon Nanotubes. When the optimal composition of the ink was used, as described earlier, the patterned dots were filled with small clusters of catalyst nanoparticles, resulting in high-quality CNTs with the desired density after CVD growth. Small variations of the order of 1-3% in composition of this optimal ink did not affect the results. Typically for optimal composition 8% and 25 % of each solvent (DMF and Glycerol respectively) was added to the master ink solution. Our task was to achieve small size iron nanoparticles in the catalyst dot clusters after drying of the ink and heat treatment of the substrate. The average size of the resulting iron nanoparticles after reduction is 6-8 nm and this parameter is essential for controlling the diameter of the subsequently grown SWCNTs. Particles less than $< 10 \text{ nm}$ size

predominately produce single walled CNTs. Fig. 1 d) shows an AFM topographic image of such a cluster of Fe nanoparticles patterned via DPN. The inset to Fig. 1 d) represents a topographic line profile (shown in red) across few nanoparticles of diameter ~ 7 -8 nm each.

After patterning by DPN, the substrates are transferred into the CVD furnace. Our CVD system is operated under ambient pressure and the flow rates of gases were controlled by calibrated mass flow controllers. Control over gas flow parameters, their concentrations, flow rates and time of the growth are important for successful growth. Methane is used as the carbon stock gas. We have tested the concept of controlling growth parameters and preconditioning the catalyst in order to see changes in overall yield of CNTs and their structural properties. The parameters that control the preferential growth of semiconducting or metallic tubes are still being investigated. However, the evidence of increased production of one type vs. the other has been indicated (Harutyunyan, 2009). Two parameters seem to play an important role: the carrier gases used during the heat treatment of Fe nanoparticles prior to growth, and the gases used during the growth. With an Ar/H₂ mixture used at the preconditioning step, the majority of the CNTs grown are believed to show semiconducting behavior. When Ar gas is substituted by He gas during the CVD process increased metallic behavior is expected. The procedure we describe below was used for growing semiconducting CNTs. Substrates were heated initially to 400 °C with a mixture of Ar/H₂ gases at flow rates of 195/175 sccm respectively for 1 hour. Next, the temperature was increased to 850 °C and the flow of Ar was switched off. The temperature was stabilized for 5 minutes with only pure H₂ gas flowing at 150 sccm for 10 minutes. To

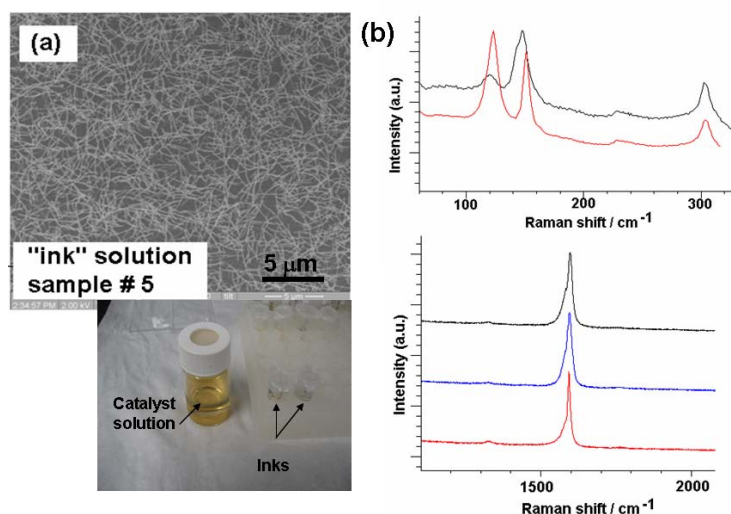


Fig. 2. SEM micrograph and Raman spectra of CNTs grown from “ink” solution spin coated on Si/SiO₂ substrate. a) Representative SEM micrograph of spin coated substrate after the CVD growth and photograph of the catalyst master and “ink” (left panel top and bottom). b) RBM (120-150 cm⁻¹) spectra and G bands peaks (~ 1600 cm⁻¹) shown on few different spots on the sample.

solution, 3-6 month. No color changes or precipitations were visually observed during these time periods.

Raman spectra were acquired on diluted CNT network samples, using the spectrometer at CNM. The curves in the top panel of Fig. 2b show two peaks at around 120 and 150 cm⁻¹, corresponding to the radial breathing mode (RBM) for two nanotubes (the peaks at 229 cm⁻¹ and 304 cm⁻¹ are Si peaks). G band peaks at ~ 1600 cm⁻¹ were also detected as seen in the bottom panel in Fig. 2b. The spectra were acquired at different locations of the same sample and show the band structure of uniform and disorder-free CNTs. The small amplitude of the D peak 1300-1400 cm⁻¹ that is typically associated with disorder indicates the high quality of our CNT material. Another indication of the purity of the CNTs can be seen in the linewidth, as the linewidth various carbon impurity D bands is much broader than nanotube D band. For SWCNTs, the D-band width will also vary with both diameter distribution and laser excitation energy. The data shown here

were acquired with an InVia Raman spectrometer using a wavelength of 633 nm for both RBMs and G band with 100x objective.

DPN patterned catalyst islands and fabrication of the CNTs devices.

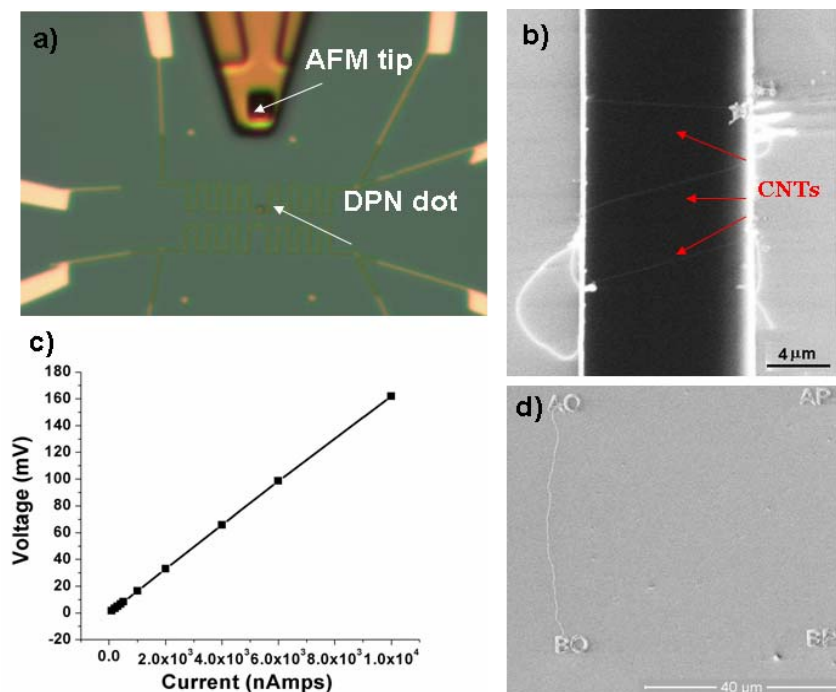


Fig. 3. DPN patterned catalyst structured at predetermined locations on the substrates. DPN patterned individual dots a) optical image with white arrows indicating AFM tip coated with “ink” top view and DPN deposited dot right after the deposition. CNTs after the growth are shown to originate from placed where DPN dots were patterned. Dots are not seen on the SEM micrographs in b) and d), however CNTs are visible. Dots were on the left side of the trench in b) and near “AO” on the d).

We currently employ two different strategies for fabrication of CNT devices. Once we have fabricated the fiducial markers, we either pattern single or few catalyst dots, or an array of dots using DPN as shown on Fig. 1. Once the DPN patterned catalyst is prepared on the substrate, the CVD synthesis takes place as described earlier. The length of the CNTs can be controlled by the growth time. Our growth time varies from 10-20 min. By adjusting the growth conditions, catalyst conditioning, heat treatment and gas mixture during preheat and actual growth, one can preferentially grow CNTs with majority of semiconducting or metallic properties. Fig. 3 b) and d) show examples where DPN isolated catalyst dots were patterned at specified locations near “AO” letter

marker and along the side of the trench (10 μm wide and 500 μm deep). In Figs. 3b and 3d, the dots themselves cannot be seen in SEM images due to poor contrast. The use of parallel DPN printing is beneficial for scaling the production of individual devices in planar 2D geometries with nanoscale precision. Alternatively, metallic leads and device structure could be prefabricated prior to DPN patterning of the catalyst dot. Fig. 3a) shows an example of such a structure. It consists of two zigzag like 500 nm wide wires which were designed to serve as on-chip thermometer devices in later measurements. The white arrow in Fig. 3a points to a DPN patterned catalyst dot which is precisely positioned in the center of one of the thermometer wires. The second white arrow points to the top view of the cantilever tip. Metals such as Cr, Ni and Pt can be used for prefabricated devices. Fig. 3a) demonstrated the precision capabilities of AFM controlled catalyst patterning that can be done with DPN techniques. In this way it will be easy to grow nanotubes across any desired structure at the last stage of fabrication. This protocol will ensure clean, contamination-free CNT embedded structures which will eventually allow us to test the limits of the performance of such CNTs based devices.

We had also recorded current-voltage curves in two terminal arrangements on one of our test samples at room temperature, as shown on Figure 3c. This particular sample exhibits a linear dependence in current vs voltage with a resistance ~16 kOhm. We had also measured the resistance of this sample in a 4 terminal arrangements. The next step is to produce and measure a series of individually contacted CNTs with various multilayered contact pads to test our theoretical predictions. Until recently it was assumed that the CVD method of carbon nanotube synthesis has limited capabilities for controllable growth of CNTs with preferential properties, orientation or diameter distribution. These limitations are still a bottleneck for

reproducibly manufacturing carbon nanotube devices. However, we believe that the recent studies done by our group and other research groups indicate exciting possibilities to overcome these challenges and allow for more economical, flexible and convenient ways to produce high quality SWCNTs with given properties.

The primary effort in this project was directed towards developing consistent protocols that would allow production and fabrication of CNT based devices with desired geometries and characteristics. We established systematic and robust procedures and methods that indicate advantages in producing consistent results for:

- 1) High quality CNT growth and characterized with Raman spectroscopy AFM techniques.
- 2) Reproducible size and geometry control.
- 3) Arrays of CNT patterned with both conventional and novel lithography techniques.
- 4) More reproducible results were obtained from DPN patterned catalyst arrays as compared to patterned catalyst using e-beam lithography.
- 5) Few representative I-V characteristics were measured.
- 6) Use of Pd as the contact metal produced reasonable transparency of the metal-nanotube contacts. Further tests are needed to test Pt as potential electrode metal.
- 7) We can prepare suspended and substrate-supported CNTs with lengths from a few to tens of microns. Additionally Raman spectroscopy studies on individual CNT indicate defect formations at CNT/Substrate interfaces. Similar observation have been reported in the literature in the past by others.
- 8) Long nanotubes and nanotube arrays can be used to fabricate a number of devices made of single SWCNTs. These structures can be used to examine devices made from individual segments and as well as to examine whether the electrical properties of individual nanotubes remain unchanged over their entire length.

Publications and presentations and other contributions

- 1) “*Synthesis and characterization of SWCNT prepared on silicon substrate with different methods of patterning catalyst particles*”, I. Kuljanishvili, O. Loh, D. Dikin, H. Espinosa, R. Piner, R. S. Ruoff and V. Chandrasekhar, March Meeting of the Am. Phys. Soc., New Orleans, Louisiana, 2008 (oral presentation).
- 2) “*CVD grown of CNTs on Si substrate from DPN patterned catalyst precursor*”, I. Kuljanishvili, D. Dikin, S. Rozhok, S. Mayle and V. Chandrasekhar, March Meeting of the Am. Phys. Soc., Pittsburg, Pennsylvania, 2009 (oral presentation).
- 3) “*Controllable Patterning and CVD Growth of Carbon Nanotubes with Direct Parallel Writing of Catalyst Ink using Dip-Pen Nanolithography*”, I. Kuljanishvili, D. Dikin, S. Rozhok, S. Mayle and V. Chandrasekhar, MRS Spring Meeting, San Francisco, California, 2010 (oral presentation)
- 4) May 2009 authors: I. Kuljanishvili, S. Rozhok, D. Dikin, V. Chandrasekhar had also filed an invention disclosure (NU#29076- High Throughput Dip Pen Nanolithography using liquid molecular catalyst and Position Controlled Synthesis of individual carbon nanotubes on a nanometer scale)
- 5) Kuljanishvili, I. Dikin D., S. Rozhok, S. Mayle, V. Chandrasekhar,: Controllable Patterning and CVD Growth of Isolated Carbon Nanotubes with Direct Parallel Writing of Catalyst Using Dip-Pen Nanolithography, *Small*, 5, 22 , 2523-2527. 2009, Cover article.

Additional references:

- 5) S. E. Shafraniuk, ‘Reversible heat flow through the carbon nanotube junctions, *EPL*, **87**, (2009) 57007
- 6) S. E. Shafraniuk, I. Kuljanishvili, *Electric Energy Generation By Arrays Of Carbon Nanotube Junctions*, Paper #: HP-007 Proceedings of The 27th Army Science

7) I. Kuljanishvili et. al. "Variations in temperature dependent Raman spectroscopy of carbon nanotubes growth with different gases during the CVD synthesis" in preparation.