

**REPORT DOCUMENTATION PAGE**

Form Approved  
OMB No. 0704-0188

The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.  
**PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.**

1. REPORT DATE (DD-MM-YYYY) 20-04-2007		2. REPORT TYPE Final Technical Report		3. DATES COVERED (From - To) 12/1/2005 - 11/30/2006	
4. TITLE AND SUBTITLE Scaled up Fabrication of High-Throughput SWNT Nanoelectronics and Nanosensor Devices				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER W911NF-06-1-0031	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Pulickel M. Ajayan				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Rensselaer Polytechnic Institute 110 8th Street, Troy, New York 12180-3590				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Army Research Office				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S) 49012.1-EG	
12. DISTRIBUTION/AVAILABILITY STATEMENT Distribution Unlimited; Approved for Public Release					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT The project's primary goal was to build integrated, on-chip carbon nanotube based devices that could be used as multi-agent sensors. The main tasks were controlled growth of carbon nanotubes of various types, for integration into the on-chip sensor, fabrication of the carbon nanotube based sensor devices and, the electrical and other relevant characterization. The project was done in collaboration with personnel at the Army Research Laboratory.					
15. SUBJECT TERMS Carbon Nanotubes, Sensors, Nanoelectronics					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES 5	19a. NAME OF RESPONSIBLE PERSON Pulickel M. Ajayan
a. REPORT	b. ABSTRACT	c. THIS PAGE			19b. TELEPHONE NUMBER (Include area code) 518-276-2322

Project Title: Scaled up Fabrication of High-Throughput SWNT Nanoelectronics and Nanosensor Devices  
Award Number: W911NF-06-1-0031  
Principal Investigator: Pulickel M. Ajayan (Professor, Rensselaer Polytechnic Institute)

## **FINAL REPORT**

### **1. Proposal Goals**

The project's primary goal was to build integrated, on-chip carbon nanotube based devices that could be used as multi-agent sensors. The main task can be divided into three sub-tasks: (a) Controlled growth of carbon nanotubes of various types, for integration into the on-chip sensor, (b) fabrication of the carbon nanotube based sensor devices and finally (c) the electrical and other relevant characterization.

### **2. Work Completed**

While pursuing the abovementioned tasks (to achieve carbon nanotube based sensor devices) we focused on extensive evaluation of the electrical transport through SWNT-based electronic devices. Our current focus is on electrical measurements through single SWNT devices at temperature ranging from the room temperature to liquid helium temperature. SWNT has generated considerable interest on one hand in nanoscale electronic device applications, due to their unique electronic properties and nanometer sizes. On the other hand, from the fundamental physics point of view, SWNTs can be treated as the ideal model systems for one-dimensional (1D) conductors with profound electron-electron interaction to influence the electronic transport through the nanotube. A brief summary of the results accomplished in the study is given below.

#### **2.1. Controlled growth of Single Wall Carbon Nanotubes**

Within the various types of carbon nanotube materials synthesis, we have been focusing mainly the controlled SWNT growth by Chemical Vapor deposition (CVD) method. We use a diblock copolymer, polystyrene-block-ferrocenylethylmethylsilane (PS-b-PFEMS) as the polymer-based catalyst for optimized SWNT growth. Spin coating a dilute solution of 1 wt % PS-b-PFEMS in toluene provides uniform ~ 100nm thick films of the catalyst. In order to get well-separated individual SWNT or SWNT rope with desired density, we have found this thickness of the catalyst layer is the optimized thickness. Finally, SWNTs are grown by the CVD process with methane flow at 920 degrees Celsius on spin-cast samples.

#### **2.2. Fabrication of SWNT-based Electronic Devices**

The next step after the growth towards the electrical transport measurements is to fabricate the nanotubes into effective electronic devices. In this section we will present the detailed fabrication procedure of single SWNT or a single bundle contained few SWNTs using state-of-the art technique that we use in our laboratory. The most commonly used standard fabrication processes include optical lithography, focused Ion beam milling and electron beam lithography techniques. Among all of these processes,

we concentrated predominantly on the electron beam lithography process to fabricate our SWNT devices. First we process bonding pads and markers on the sample with SWNTs on it by electron beam lithography followed by a deposition of Ti/Au using electron beam evaporator. Next, we find SWNTs in scanning electron microscope (SEM) or in atomic force microscope (AFM) and get the coordinates of the position of the SWNT with respect to the nearest marker. Finally, with one step left for electron beam lithography and electron beam deposition, we fabricate the electrical metal contacts on SWNT.

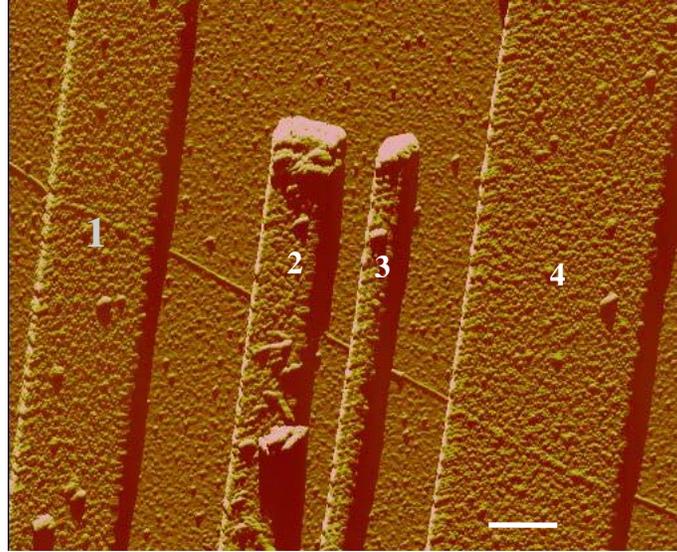


Figure1. AFM image of a SWNT device with four electrodes. The scale bar corresponds to 500nm.

Final contacts are made of a thin layer of Ti  $\sim$  2-5nm and a Au layer of about 50-100nm on top of the Ti layer. Our device geometry is shown in the figure 1. In order to perform 2-terminal and four terminal measurements on one single device, we fabricate four identical contacts on each SWNT. In this identical contact design, we compare the electronic characteristics of each segment between any two leads from the single device. In other cases, we fabricate the four contacts with different shapes and different electrode spacing as shown in figure1. The different shapes and different spacing between the electrodes additionally allow us to study three different 2-terminal devices from each single device as in the previous case for identical contacts. In this case, as one can study the effects of the electrode's dimension on the electronic tunnel spectra similarly, the fundamental studies in the frame of quantum interference can be studied with varying electrodes spacing.

### 2.3. Room Temperature Current-Voltage Measurements on SWNT Devices

In this section we present some of the results on room temperature electrical measurements of our devices. We have performed two-terminal Current-Voltage (I-V) measurements at room temperature on some of our SWNT devices in the geometry

shown in figure.1 with different electrode spacings and dimensions. Figure 2(a) displays the I-V characteristics of one such device. Three different curves show the I-V measurements between the leads 1 and 2 (Leads<sub>1-2</sub>), between leads 2 and 3 (Leads<sub>2-3</sub>) and between leads 3 and 4 (Leads<sub>3-4</sub>) from same device. It is very clear from the graph that the electronic transport depends on the dimension of the contact area and also with the distance between the leads. By introducing asymmetric tunnel barrier using the different shape we plan to study the electrical characteristics at higher bias voltage in order to probe the asymmetric tunneling at non-linear regime. By this approach, we can study the tunneling density of states of nanotubes at the contact region at low temperatures.

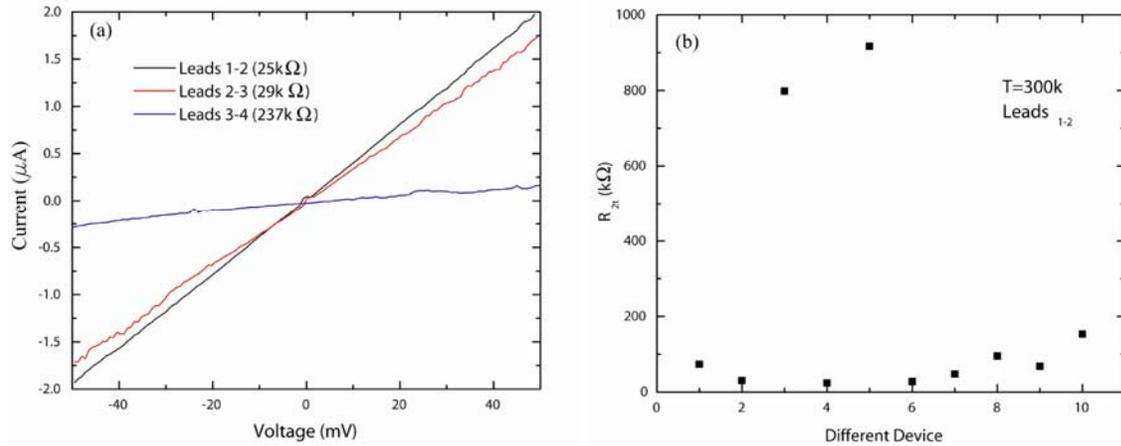


Figure 2. Room temperature I-V measurements. (a) two-terminal I-V measurements between three sets of leads for one single device, (b) two-terminal resistance between leads 1 and 2 for 10 different devices.

The most challenging part towards clean electrical measurements through these devices is in reducing the contact resistances. Due to the high contact resistance between the SWNT and the leads, it is very difficult to perform very sensitive measurements where electrical signal through the device is almost comparable with the instrument noise. In order to compare the contact resistance from our devices with the previous works in this field from other groups, we have measured the two-terminal contact resistance from 10 separate devices between leads 1 and 2 (Leads<sub>1-2</sub>). The corresponding result is presented in figure 2(b). From this figure, out of 10 devices for about 8, the two terminal resistance at room temperature is around  $100\text{k}\Omega$  which is very good compared to other reported work on SWNT devices. Relatively low contact resistance provides us model one-dimensional systems to observe various interesting and important quantum mechanical phenomena in these devices.

### 3. Conclusions and Ongoing Work

In the past year, we have been able to optimize the controlled growth of SWNTs for device applications as well as for fundamental studies. Successful fabrication techniques have been achieved in our lab using electron beam lithography and electron beam deposition to fabricate electrical contacts on a single SWNT or on an individual bundle of SWNTs. The promising results in the view of the contact resistance between SWNTs and

the metallic contacts open many avenues towards the electrical characterization at different regimes, different temperatures, and different electric and magnetic fields.

Due to strong electron-electron interaction and profound electronic confinement around the circumference, a large spacing between 1D sub-bands can be achieved by reducing the length of the nanotube between the leads. We expect to observe this confinement effects even at room temperature. In this direction, we aim to fabricate the devices with least possible electrode separation. We have found in AFM investigation, SWNTs are in bundles of few (3-4) single SWNTs and it obviously reduces the confinement effects. Our next goal is to find an efficient way to separate the SWNTs from the bundles in order to study isolated SWNT based electronic devices. One way to approach towards single SWNT device, we plan to burn some of the SWNTs from the bundles by passing high current through the devices.

Finally, some of the bundles get burned resulting in open electrical circuits with a gap between the two broken segments of the bundle. Once we have optimized this process to control the gap (nanoscale) introduced into individual nanotubes or bundles, via electrical burning, we plan to attach molecules between these gaps and study their molecular transport behavior. This would allow sensing of various species, by making the gap ends (tips of broken tubes) selective to absorption of only certain molecular species. Here nanotubes would behave as the effective electrodes, with nanoscale gaps. Controlling the gap between the nanotubes different sized molecule can be studied through the electronic transport through them.

#### **4. Publications and Presentations**

##### *Peer-reviewed publications:*

- i) Sumanjeet Kaur, Sangeeta Sahoo, Pulickel Ajayan and Ravi S. Kane, “Capillarity-driven assembly of carbon nanotubes on substrates into dense vertically aligned arrays” (submitted).
- ii) Sangeetha Sahoo, Sarah Lastella, G. Mallick, S. Karna and Pulickel Ajayan, “Controlled electrical burning of nanoscale gapes in carbon nanotube bundles” (to be submitted).

##### *Select invited talks by the P (Pulickel M. Ajayan):*

- i) “Engineering of Carbon Nanotube Architectures”, Seminar, California Institute of Technology, Pasadena, CA (February 22, 2006).
- ii) “Controlled Assembly of Carbon Nanotube Structures”, Brandon Symposium, TMS Meeting, San Antonio, TX (March 13, 2006).
- iii) “Engineering of Carbon Nanotube Architectures”, NSLS/CFN User Meeting, Brookhaven National Laboratory, Upton, NY (May 15, 2006).
- iv) “Engineering of Carbon Nanotube Architectures”, Missouri Nanoalliance Meeting, University of Missouri, Columbia, MO (October 7, 2006).
- v) “Engineering of Carbon Nanotube Architectures”, Workshop on Carbon Technologies, Pennsylvania State University, State College, PA (October 16, 2006).
- vi) “Engineering of Carbon Nanotube Architectures”, Kavli US-Chinese National Academy of Science Meeting, Irvine, CA (October 27, 2006).

- vii) “Engineering of Carbon Nanotube Architectures”, Seminar, Rutgers University, New Brunswick, NJ (November 17, 2006).
- viii) “Engineering of Carbon Nanotube Architectures”, Seminar, University of Pennsylvania, Philadelphia, PA (November 16, 2006).
- ix) “Carbon Nanotube Architectures and Applications” Distinguished Lecture Series, Northeastern University, Boston, MA (November 28, 2006).
- x) “Engineering of Carbon Nanotubes” Symposium G, Materials Research Society Fall Meeting, Boston, MA (November 29, 2006).
- xi) “Engineering of Carbon Nanotubes” Gordon Research Conference on Chemistry of Hydrocarbon Resources, Venture, CA (January 9, 2007).
- xii) “Engineering of Carbon Nanotubes” Special seminar, Applied Materials, San Jose, CA (January 11, 2007).

### **5. Personnel and Collaboration with DoD Laboratory**

The project was directed by the PI, Prof. Pulckel M. Ajayan. A post-doctoral fellow, Dr. Sangeetha Sahoo was the main personnel working on the project. A student, Sarah Lastella, collaborated on the project, in particular in the development of the polymer catalyst for nanotube growth. Several of the facilities that are part of the NSF Nanoscale Science and Engineering Center (NSEC) and the New York Focus Center for Interconnects, contributed to the project.

A strong collaboration was established between the Army Research Laboratories, between Dr. Shashi Karna and Dr. Govind Mallick. The collaboration involved exchange of specimens, discussions, visits and planning of experiments. Some of the future publications that will come out of the initial results obtained as part of the project will involve personnel from both institutions. The future planned work will also be based on collaboration between RPI and ARL.