9-16-2005

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Matthew R. Maschmann  
*Mechanical Engineering, Birck Nanotechnology Center, Purdue University*

Placidus B. Amama  
*Mechanical Engineering, Birck Nanotechnology Center, Purdue University*

Amit Goyal  
*Department of Chemistry, New Jersey Institute of Technology*

Zafar Iqbal  
*Department of Chemistry, New Jersey Institute of Technology*

Roy Gat  
*Sekitechnotron USA*

*See next page for additional authors*

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Parametric study of synthesis conditions in plasma-enhanced CVD of high-quality single-walled carbon nanotubes

Matthew R. Maschmann a,b, Placidus B. Amama b, Amit Goyal c, Zafar Iqbal c, Roy Gat d, Timothy S. Fisher a,b,*

a Purdue University, School of Mechanical Engineering, West Lafayette, IN 47904, USA
b Purdue University, Birck Nanotechnology Center, West Lafayette, IN 47904, USA
c New Jersey Institute of Technology, Department of Chemistry, Newark, NJ 07102, USA
d Sekitechtron USA, 1153 Bordeaux Dr #102, Sunnyvale, CA 94089, USA

Received 5 April 2005; accepted 16 July 2005
Available online 16 September 2005

Abstract

High-quality single-walled carbon nanotubes (SWCNTs) have been synthesized from H2–CH4 mixtures on a MgO-supported bimetallic Mo/Co catalyst using microwave plasma-enhanced chemical vapor deposition (PECVD). Reaction parameters including temperature, H2:CH4 ratio, plasma power, and synthesis time have been examined to assess their influence on SWCNT synthesis. Raman spectroscopy and high-resolution field emission scanning electron microscopy reveal that the quality, selectivity, density and predominant diameter of SWCNTs depend on the varied synthesis parameters. Results of this study can be used to optimize SWCNT synthesis conditions and products and to improve understanding of the growth of SWCNTs by PECVD.

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Keywords: Carbon nanotubes; Plasma deposition; Raman spectroscopy

1. Introduction

Single-walled carbon nanotubes (SWCNTs) possess an unparalleled combination of mechanical strength and transport characteristics, and great interest exists for their use in a variety of applications including nanoscale electronic devices [1,2], compact X-ray cathodes [3], and composite materials [4], among others. The ultimate impact of SWCNTs in many engineering applications, however, depends on precise control of their physical properties and, as such, the synthesis environment in which they grow. Plasma-enhanced chemical vapor deposition (PECVD) is an attractive method for carbon nanotube (CNT) synthesis because of its independent control of synthesis variables such as substrate temperature, local gas composition, plasma power density, and operating pressure. The dissociation of hydrocarbons in plasma has been suggested as a mechanism to allow synthesis of CNTs and carbon nanofibers (CNFs) at low temperatures and to control gas chemistry near the growth substrate [5,6]. In addition, vertical alignment of CNTs due to plasma sheath effects rather than spatial confinement have been well documented [7–9] and could aid in development of devices requiring high-spatial density of components, such as ultra dense digital memory.

The synthesis of SWCNTs using PECVD is a new technique with relatively little known about the influence of synthesis parameters on SWCNT growth. CNTs grown by PECVD have traditionally been limited to the production of multi-walled carbon nanotubes...
(MWCNTs). Recently, however, SWCNT synthesis has been accomplished using radio-frequency PECVD [10–13] and shielded microwave PECVD [14]. Among these reports, synthesis conditions vary substantially, and changes in SWCNT properties as a result of the parameter changes are not quantified. To date, little emphasis has been placed on optimizing the influential synthesis parameters that affect quality and growth of the SWCNTs in a plasma environment. Realizing the unique advantages offered by PECVD, a systematic parametric study of this nature, such as that performed for double-walled CNTs grown by thermal CVD [15], provides insights that could greatly enhance the quality of SWCNTs and enable the control of their properties (e.g., density, alignment, length and diameter).

In this report, a comprehensive parametric study of the synthesis parameters affecting the growth of SWCNTs using microwave PECVD and an MgO-supported Mo/Co catalyst. These parameters include the synthesis temperature, inlet feedstock gas composition, growth time, and plasma power. Samples obtained after reaction are characterized using field emission scanning electron microscopy (FESEM), Raman spectroscopy, transmission electron microscopy (TEM), and atomic force microscopy (AFM). The results indicate that SWCNT selectivity can potentially be controlled by selection of synthesis parameters.

2. Experimental details

Carbon nanotube synthesis was conducted in a SEKI AX5200S microwave PECVD reactor, shown in Fig. 1. A 1.5 kW (2.45 GHz) ASTeX AX2100 microwave generator supplied power to the plasma, while inductive substrate heating was supplied by a 3.5 kW radio-frequency power supply acting on a graphite susceptor. The reactor walls were electrically grounded and water cooled. The growth substrate rested on a 5.1 cm diameter, 3.3 mm thick molybdenum puck used to concentrate the plasma directly above the sample. A Williamson dual wavelength pyrometer (model 90) monitored the surface temperature of the substrate, while a shielded K-type thermocouple located 2.5 mm below the surface of the heated graphite susceptor allowed for temperature feedback control. A main panel was used to control gas flow rates, chamber pressure, substrate temperature and microwave plasma power.

The catalyst and support were prepared by a wet mechanical mixing and combustion synthesis procedure. This catalyst has been developed for scaled-up growth of SWCNTs using a carbon monoxide thermal CVD process [16]. A solution of molybdenum and cobalt nitrate hexahydrate was prepared in atomic ratio of 4:1, and several drops of this solution were added to a solution containing 2–3 g of magnesium nitrate and 0.50 g of citric acid. Distilled water was then added to obtain a translucent pink Co(NO3)2Æ6H2O solution. Citric acid was used as a foaming agent. The solution was placed in a ceramic boat covered by a flexible aluminum sheet, placed inside a quartz tube and loaded into a furnace held at 550 °C for 5 min. The temperature was controlled using a programmable logic controller integrated with the furnace. The system was then allowed to cool to room ambient. The resulting bimetallic Mo/Co catalyst on nanoporous MgO-support was ground to a fine powder using a mortar and pestle, resulting in particles ranging from hundreds of nanometers to tens of microns in size. This catalyst and support system has been shown to play a role in controlling SWCNT diameter, as suggested by Liu et al. [17]. A mass of 0.030 g of catalyst powder was added to 25 ml of acetone, and the suspension was sonicated for 3 h. Approximately 10 drops of the catalyst suspension were then immediately dispensed onto a silicon wafer using a glass pipette. The suspension was allowed to dry for 30 min in air before the substrate was inserted into the PECVD reactor.

Prior to synthesis, the reactor chamber was slowly evacuated to a pressure of 0.5 Torr by an external mechanical pump. Hydrogen (Praxair 4.5 grade) gas was then introduced into the chamber, maintained at approximately 0.6 Torr, at a flow rate of 50 sccm for 5 min to purge the chamber. The chamber was then pressurized and maintained at 10 Torr with hydrogen gas at a flow rate of 50 sccm. Induction substrate heating was applied until the synthesis temperature was reached. Upon stabilization at the synthesis temperature, a hydrogen plasma was introduced at a power determined by the parametric study. Methane (Praxair ultra high purity) was then introduced into the chamber, initiating the synthesis process. Various synthesis times and inlet gas flow rates were employed for the parametric study. After terminating CNT synthesis, the reactor was evacuated, and the sample was allowed to cool to room temperature before being removed from the microwave plasma.
Nitrogen gas (Praxair 4.8 grade) was used to purge and pressurize the chamber. The parametric study focused on the effect of synthesis temperature, inlet gas composition, synthesis time, and plasma power on the SWCNT growth. Synthesis of SWCNTs is expected to be favored in a narrow temperature range, and as such temperature was the initial variable studied. Because the heated susceptor temperature is determined directly through feedback control, increments in this temperature were used rather than the substrate surface temperature measured by the pyrometer. This study is the first to report the use of a (dual wavelength) pyrometer to measure the catalytic substrate surface temperature during PECVD synthesis of SWCNTs. Our results indicate that plasma power and susceptor temperature strongly influence the temperature difference between the catalytic surface and heated susceptor, with differences of greater than 100 °C observed in this study. Previous experience with this reactor has shown that the substrate surface temperature may be either substantially higher than the susceptor temperature when using a high-power plasma or lower than the susceptor temperature for low-power plasma. Other studies [10–14] on synthesis of SWCNTs by PECVD measured temperature beneath the catalytic substrate by means of a thermocouple, and it is unclear whether the reactive substrate surface was at a higher or lower temperature than that measured by thermocouple.

Raman spectra were recorded and averaged over five locations on each sample. A near-infrared system with cooled CCD (charge coupled device) detection and 632.8 nm wavelength laser excitation were used for recording the Raman spectra. A Hitachi S-4800 FESEM instrument imaged the SWCNT product for length, density, and diameter estimates for tube bundles. A JEOL JEM-2000FX TEM and a Veeco DI Dimension 3100 AFM in tapping mode were also used to determine relative density and diameter of the synthesized SWCNTs.

3. Results and discussion

3.1. Effect of temperature

For this study, susceptor temperatures of 600, 700, 800, 900 and 950 °C, were investigated with corresponding top substrate surface temperatures of 580, 665, 710, 770 and 823 °C, as measured by the pyrometer. The synthesis parameters that were held constant during the temperature variation included an operating pressure of 10 Torr, 200 W microwave plasma power, and mass flow rates of 50 sccm hydrogen and 5 sccm of methane for 20 min. These conditions were chosen based on previous successful SWCNT synthesis studies carried out using this catalyst and are to be considered standard conditions unless otherwise stated.

The Raman spectra for the carbon products obtained from samples grown at the foregoing temperatures are shown in Fig. 2. The spectra reveal a distinct peak near 1580 cm⁻¹ that is associated with highly ordered graphite (called the G-band) and another peak at 1320 cm⁻¹ (called the D-band) associated with sp²-bonded nanocrystalline or amorphous carbon species for all temperatures studied. Radial breathing mode (RBM) peaks found at Raman shifts below 400 cm⁻¹, indicative of SWCNTs, are observed for carbon products synthesized at susceptor temperatures of 800 °C and higher, suggesting that 800 °C is the threshold for SWCNT synthesis under the standard conditions. The products from 800°C growth exhibit a RBM peak at 214 cm⁻¹, corresponding to a diameter of 1.1 nm according to the relation \[ \omega_{\text{RBM}} = 12.5 + 223.5/d(\text{nm}) \] [18] for SWCNT bundles. The products from the sample grown at a susceptor temperature of 900 °C exhibit a strong RBM peak at 214 cm⁻¹ and less intense shoulders at 190 and 250 cm⁻¹, corresponding to SWCNT diameters of 1.1, 1.3, and 0.9 nm, respectively. For SWCNTs grown at a susceptor temperature of 950 °C, the most intense RBM peak shifts to 190 cm⁻¹ with less intense peaks at 214 and 250 cm⁻¹. The shift in the most intense RBM from 214 cm⁻¹ at a susceptor temperature of 800 °C to 190 cm⁻¹ at 900 °C suggests that synthesis of larger-diameter SWCNTs are preferred at higher temperatures. For nanoscale catalyst particles, a decrease in particle size leads to an exponential increase in carbon solubility [19]. At lower synthesis temperatures, small catalyst particles are able to support higher carbon content than larger particles, and these particles may then be able to precipitate SWCNTs with less thermal energy than larger particles. As synthesis temperature increases, larger-diameter catalyst particles are expected to become catalytically active. The apparent preferential growth of larger-diameter SWCNTs with increasing temperature,
however, contradicts theoretical energy studies, which predict that smaller-diameter SWCNTs require higher formation energies [20].

The ratios of the G- to D-band intensity, which indicate the relative amount of amorphous carbon and sidewall and tube end defects compared to highly ordered graphitic carbon, have been used as a purity index to assess the purity of the SWCNTs synthesized [21–23]. A G- to D-band ratio much greater than unity indicates an abundance of highly ordered graphite and graphitic defects and a low amount of undesired carbon species. As shown in Fig. 2, samples grown at 900 and 950 °C exhibit the highest G to D ratio (greater than 6.5), while the sample grown at 800 °C exhibits a low G to D ratio of approximately 3. Carbon deposited at susceptor temperatures of 700 and 600 °C do not exhibit RBM peaks and have G- to D-band intensity ratios near unity. In the absence of catalytic activity, self-pyrolysis of hydrocarbon species and radicals produced in the plasma creates relatively large amounts of amorphous carbon deposition and can explain the low quality of these samples.

FESEM micrographs in Fig. 3 show that a relatively low density of SWCNTs is grown at a susceptor temperature of 800 °C, with typical lengths of hundreds of nanometers. A much higher density of SWCNTs was observed for samples grown at susceptor temperatures of 900 and 950 °C, often forming large-diameter bundles of 5 nm or greater with lengths up to 3 μm. Similar types of preferential temperature windows for optimal SWCNT growth have been reported for thermal CVD synthesis [24]. At a susceptor temperature of 700 °C, sparse populations of small-diameter CNTs were observed by FESEM, while no CNTs were observed for the 600 °C sample. As mentioned previously, no RBM peaks were observed for the sample grown at a susceptor temperature of 700 °C, suggesting that the population of CNTs, if SWCNTs, was either too sparse for detection or consisted of small-diameter MWCNTs.

3.2. Effect of inlet gas composition

As shown in the results of temperature variation, dense SWCNTs of similar diameters and quality were grown at susceptor temperatures of 900 and 950 °C. Based on the purity index, the intensity of the RBMs with respect to the G-band, and the density of SWCNT growth as determined by FESEM, the optimum synthesis temperature was chosen to be 900 °C, and this temperature was fixed in the parametric study of inlet gas composition. Hydrogen to methane volumetric flow rates of 50:2, 50:5, 50:10 and 30:10 (all measured in sccm) were introduced at otherwise standard synthesis conditions. For all samples, Raman spectra of various gas compositions shown in Fig. 4 reveal two main active RBMs at 190 and 214 cm⁻¹ with a shoulder at 250 cm⁻¹ corresponding to SWCNT diameters of 1.3, 1.1, and 0.9 nm, respectively. SWCNTs grown in the most carbon lean environment (50:2) exhibit two equally intense RBM peaks at 190 and 214 cm⁻¹, while SWCNTs grown in the most carbon rich environment (30:10) exhibit a strong peak at 214 cm⁻¹ with only a faint shoulder at 190 cm⁻¹. This shift suggests that smaller-diameter SWCNTs are selectively favored in the carbon rich environment, with larger-diameter SWCNTs equally

![Fig. 3. FESEM micrographs showing effect of susceptor temperature. (a) 700 °C, (b) 800 °C, (c) 900 °C, (d) 950 °C. Scale bar = 100 nm.](image-url)
populated in carbon lean environments. A transition to the selectivity of smaller-diameter SWCNTs with increased methane composition can be observed from intermediate gas compositions. Examination of the normalized G- and D-band intensities in Fig. 4 show that all samples display approximately the same quality. Higher methane concentration in the inlet gas mixture did not increase the relative amount of amorphous carbon formation.

FESEM micrographs shown in Fig. 4 reveal that the density and length of SWCNTs increase as the ratio of methane incrementally increases from 50:2 to 50:10. A further increase in the methane inlet composition from 50:10 to 30:10 resulted in no observable increase in density. Relatively sparse SWCNTs forming thin bundles with diameters less than 5 nm were observed for the sample grown with a 50:2 gas composition. Gas compositions of 50:5 and 50:10 resulted in large bundles of SWCNTs with diameters up to 10 nm, and with lengths of up to several microns at the 50:5 gas composition and up to 12 μm at the 50:10 gas composition. SWCNT products of comparable length, bundle diameter, and density exist between samples grown at 30:10 and 50:10 gas compositions. Fig. 6 shows a representative TEM micrograph of a SWCNT bundle grown with the 50:10 gas composition. The increase in catalytic activity with increasing methane composition from 50:2 to 50:10 suggests that diffusion of carbon through the catalyst in this synthesis regime is a source-dependent phenomenon. SWCNT density is similar between samples grown at 50:10 to 30:10 gas compositions, indicating that the carbon supply mode has shifted from source-dependent to rate-dependent. No increase in the amount of amorphous carbon was observed, indicating that atomic hydrogen generated within the plasma may be effective at removing amorphous carbon deposits, as has been suggested by Meyyappan et al. [8].

3.3. Effect of synthesis time

The plasma synthesis environment contains energetic ions that could potentially degrade or destroy SWCNTs for extended synthesis times. For SWCNTs to be useful for integration into applications, they must retain high quality even for extended synthesis times. Previous studies have shown that short synthesis times produce high-quality SWCNTs with little amorphous carbon in both thermal CVD [22,25] and PECVD [13]. Extensive Raman spectroscopy and FESEM studies have revealed that the catalyst used in this study exhibited a lifetime of several hours, without SWCNTs transforming to large-diameter MWCNTs as reported for a different catalyst in a PECVD process [13]. Synthesis times examined in this study included 1, 5, 20, 60, and 180 min under otherwise standard synthesis conditions.

Fig. 5 shows the Raman spectra for SWCNTs produced under various synthesis times. RBM peaks were again observed at 190 and 214 cm⁻¹ with a faint shoulder at 250 cm⁻¹ for all samples, corresponding to SWCNT diameters of 1.3, 1.1, and 0.9 nm, respectively. At short synthesis times, the dominant RBM peak occurred at 190 cm⁻¹, but samples grown for 20 min and
greater exhibited a dominant RBM peak at 214 cm\(^{-1}\), with decreased intensity of the 190 cm\(^{-1}\) peak with increased synthesis time. The shift in dominant RBM peaks indicates either that smaller-diameter SWCNTs remain catalytically active longer than larger diameter SWCNTs, or that larger diameter SWCNTs are more susceptible to damage by energetic ions and are eliminated.

Examination of the G- to D-band in Fig. 5 reveals that samples synthesized for 1 min resulted in the highest purity index, while those grown for 180 min showed the poorest purity index. Synthesis times of 20 and 60 min resulted in nearly identical purity indices, half that of the 1 min synthesis yet roughly three times greater than that of the 180 min synthesis. It is common for catalyst activity to decrease with synthesis time because of gradual catalyst poisoning, leading to reduced quality and increased amorphous carbon deposition \cite{23,26}. It is also possible that increased exposure to energetic ion collisions produces lattice imperfections within SWCNTs, leading to a more intense disordered carbon peak.

Extensive FESEM investigation found that SWCNT density generally increased with synthesis time. Fig. 5 shows sparse populations of SWCNT bundles at a synthesis time of 5 min. It is expected that an increase in synthesis time would result in longer SWCNTs with increased interactions among the tubes, resulting in larger bundles. While large bundles of SWCNTs are clearly visible in FESEM images, individual SWCNTs are difficult to resolve. For samples synthesized for 5 min, sparse bundles having diameters of less than 5 nm were observed with lengths of hundreds of nanometers to one micron. Larger diameter bundles became more numerous at synthesis times of 20 min or greater. SWCNTs synthesized for 60 min resulted in randomly oriented bundles with some straight bundles reaching lengths of up to 5 \(\mu\)m and having diameters from 3 to 30 nm. A synthesis time of 180 min resulted in many straight bundles of SWCNTs of up to 15 \(\mu\)m in length with diameters of up to 30 nm, although the spatial density of the bundles decreased slightly. It can also be seen in Fig. 5 that a synthesis time of 180 min created dimples and irregularities on the top surface of the silicon substrate, demonstrating the resilience of SWCNTs in a plasma environment. The sample synthesized for 60 min was further examined using an AFM in tapping mode. The
AFM image in Fig. 7 shows both individual SWCNTs, 1.3 nm in diameter, and SWCNT bundles of diameter 2.5 and 6.3 nm, in general agreement with information gained from Raman spectroscopy and FESEM micrographs.

3.4. Effect of plasma power

Plasma power is a variable that can significantly alter the synthesis environment. Plasma power density varies greatly among reactors, and therefore plasma power input is not directly comparable from system to system. Relative changes in volumetric plasma power density, however, are expected to show more comparable results among different reactors. For our microwave PECVD reactor, a plasma ball of approximately 2 in. in diameter is generated directly above the catalytic substrate, and little change in plasma volume was observed for the plasma powers considered. Therefore, an increase in input plasma power produced a roughly proportional increase in power density. Plasma power was varied from 130 to 500 W, which represent the lower and upper limits for safe plasma generation at 10 Torr for the reactor. The plasma power density plays a direct role in heating the top substrate surface, with plasma powers of 130, 200, 350, and 500 W generating surface temperatures of 760, 770, 800, and 814 °C, respectively, for a fixed susceptor temperature of 900 °C. In addition to altering the surface temperature, gas composition within the reactor is altered with changes in plasma power density.

Methane is used extensively for SWCNT synthesis in thermal CVD because of its stability at high temperatures [22–24,27]. A theoretical model of a high-power
density hydrogen and methane plasma \cite{8} and similar model for the reactor used in this study \cite{28} show that methane is highly decomposed for the plasma powers studied herein, with a two order of magnitude reduction in molar percentage between plasma powers of 130 and 500 W. These prior models also indicate that the concentration of acetylene (C$_2$H$_2$) remains nearly steady but declines slightly with increasing plasma power and is the second or third most abundant component, depending on plasma power.

Raman spectroscopy reveals RBM peaks located at 190 and 214 cm$^{-1}$ and a shoulder at 250 cm$^{-1}$ for all plasma powers considered in the study. No noticeable changes in dominant RBM peaks are observed among the samples grown at various plasma powers. Fig. 8 shows that the relative intensity of the normalized G and D bands for all plasma powers are similar, with the sample synthesized at a 500 W plasma displaying slightly lower quality. FESEM micrographs reveal that the sample grown at 130 W exhibits much lower spatial density of SWCNTs compared to higher energy plasmas. A sample grown without plasma (thermal CVD, not shown here) at otherwise standard conditions resulted in no observable SWCNTs or MWCNTs by FESEM or Raman spectroscopy. A general increase in density occurs with increasing plasma for the entire range of plasma powers examined in this study, with the most noticeable increase in density occurring between samples grown at 130 and 200 W and between 200 and 350 W, as seen in Fig. 8. Bundles of SWCNTs grew to a length of 5 $\mu$m with bundle diameters from 5 to 30 nm for the 500 W case. It is important to note that the sample synthesized at 500 W displayed a higher density of SWCNTs and lower surface temperature than the sample synthesized at a susceptor temperature of 950 $^\circ$C and standard plasma power of 200 W, indicating that the increase in catalytic activity is not a result of increased thermal energy. Nor can the activity be attributed to an increased concentration of acetylene or methane. The increased catalytic activity is therefore attributed to either the increase in a hydrocarbon species other than acetylene or methane, or, alternatively, to higher energy atomic hydrogen ions that more effectively remove amorphous carbon from catalyst surfaces.

4. Summary and conclusions

A systematic variation of important SWCNT synthesis parameters was performed utilizing a Mo/Co catalyst embedded in an MgO-support using a microwave PECVD reactor. SWCNT density, length and diameter were shown to be sensitive to all synthesis conditions varied in this study. Temperatures of the catalytic surface and the heated susceptor were monitored, with significant differences observed between the two. A temperature window for dense SWCNT activity was found to exist between susceptor temperatures of 800–950 $^\circ$C, corresponding to catalytic surface temperatures of 710–823 $^\circ$C, with an optimum quality and SWCNT density found at a susceptor temperature of 900 $^\circ$C (770 $^\circ$C catalytic surface temperature). Low SWCNT density was observed at synthesis temperatures below the optimum temperature, with a slight decrease in quality at higher temperatures. The volumetric flow rate of hydrogen to methane was found to be optimal at a ratio of 50:10, with a lower density of SWCNTs observed above and below this ratio. SWCNT quality remained nearly constant for all gas composition ratios studied, suggesting that atomic hydrogen produced in the plasma is effective at removing amorphous carbon. The catalyst remained active at 180 min of synthesis, with SWCNT bundle length and density increasing with time. High-purity SWCNTs were produced at a synthesis time of 1 min. Increased plasma power density was observed to enhance SWCNT density, with little effect on purity.

Raman spectroscopy revealed that a change in synthesis conditions alters diameter selectivity as well. Consistent RBM peaks, characteristic of SWCNTs, were observed for samples synthesized at various synthesis conditions, but shifts in the most intense peaks, and thus most abundant SWCNT diameters, were favored for given parameter selections. Smaller diameter tubes were shown to favor low temperature, high-methane inlet gas composition ratio, and long synthesis times. Samples grown at various plasma powers exhibited no noticeable shift in dominant RBM peaks. These observations are expected to significantly enhance the understanding and lead to better control of SWCNT synthesis using PECVD.

Acknowledgements

Purdue authors acknowledge funding from the NASA-Purdue Institute for Nanoelectronics and Computing and the Birck Nanotechnology Center in support of this work, Mr. Jun Xu and Dr. Xi Zhang for TEM and AFM assistance, as well as Dr. Hartmut Hedderich for his assistance and expertise with Raman spectroscopy. NJIT authors acknowledge US Army and NSF grants in support of this work.

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