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## LDRD Final Report on Carbon Nanotube Composites

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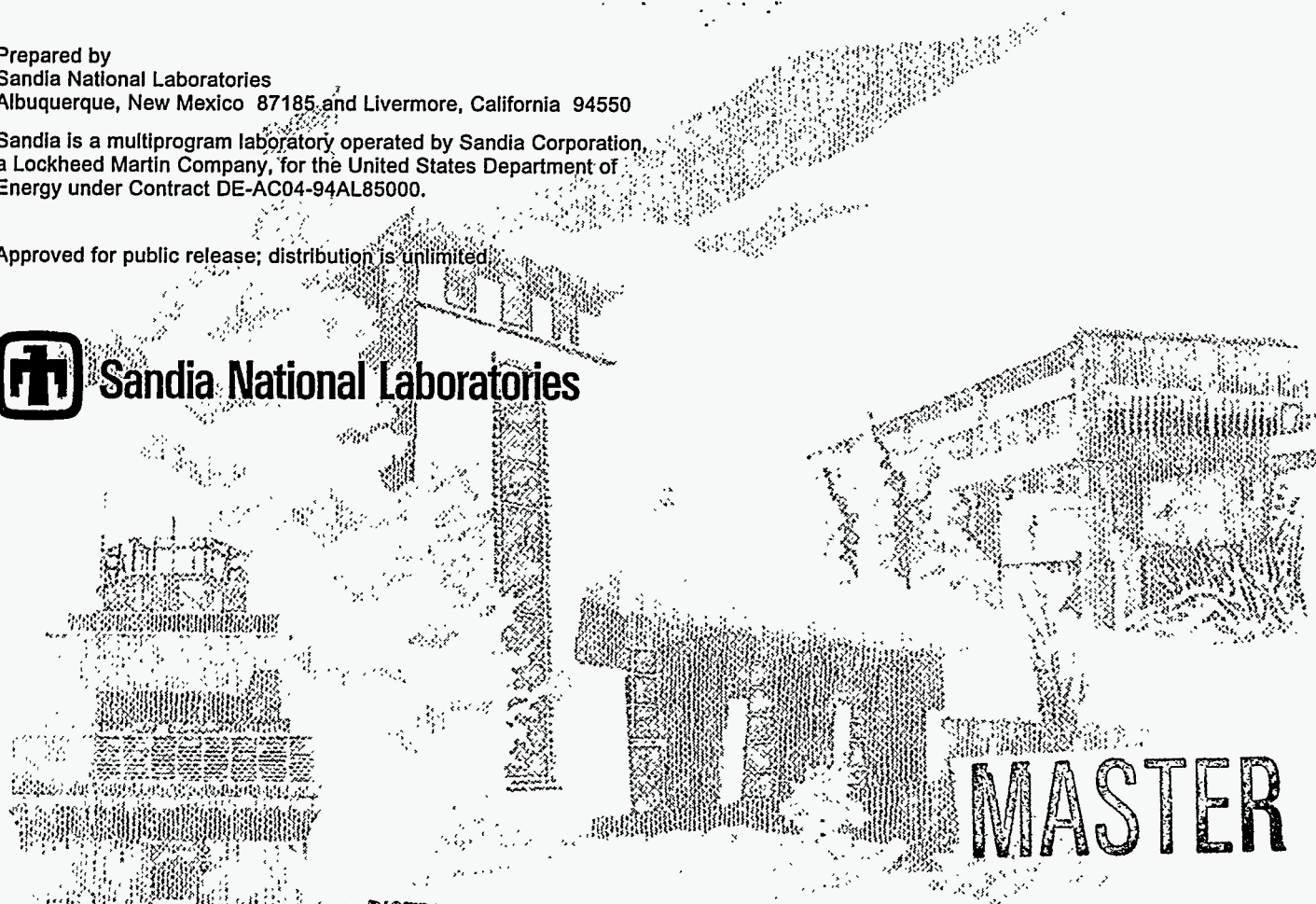
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## **LDRD Final Report on Carbon Nanotube Composites**

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### **Abstract**

Carbon nanotubes and their composites were examined using computational and experimental techniques in order to modify the mechanical and electrical properties of resins. Single walled nanotubes were the focus of the first year effort; however, sufficient quantities of high purity single walled nanotubes could not be obtained for mechanical property investigations. The unusually high electrical conductivity of composites loaded with <1% of multiwalled nanotubes is useful, and is the focus of continuing, externally funded, research.

# LDRD Final Report on Carbon Nanotubes and Their Composites

## Contents

Introduction	2
Results and Discussion	3
Continuing Research	6
Conclusions	6
Abstracts of Published Manuscripts	7
Abstracts of Accepted Manuscripts	8
Acknowledgments	8
Distribution	9

## Introduction

This LDRD (Laboratory Directed Research and Development) project was funded for two years beginning in October 1994 (FY93) and was designed to investigate the properties of composites made from a new form of carbon: carbon nanotubes. These nanotubes range in diameter from 10 Ångstroms (0.1 nm) to as much as 100 nm and are, in essence, rolls of graphite. The tubes may be composed of a single graphitic wall, a spiral-wrapped roll, or most commonly, nested or multiwalled tubes. The tubes are commercially available from several sources; however, the processes used to make the tubes varies enormously.

### Carbon Nanotube Growth and Production

All the nanotubes investigated in this project were obtained from commercial sources. The cost of nanotubes varies enormously: from tens of dollars/gram for impure, electric arc generated single walled material (which may yield <1% nanotubes) to as low as \$30-40/pound for high purity, vapor phase grown multiwalled material.

A metal-catalyzed electric arc process is used for the synthesis of single walled nanotubes. The catalyst is generally a mixture of first row transition metals which is mixed with finely divided carbon blacks. The resulting mixture is then pressed into a rod that serves as an electrode. As an electric arc consumes the electrode, metal nanoparticles are formed from which a single walled nanotube grows. The metals stabilize the open end of the nanotube, which otherwise would close to a fullerene-like cap. The yield of nanotubes is very low (few percent at best); the majority of the material is amorphous and glassy carbons along with a large amount of metal particles that are encapsulated by carbon. Single wall tubes can also be grown by a laser process, but such materials are not commercially available.

Multiwalled tubes can be prepared by a similar electric arc process, but a catalyst is not required. In this case, the growing tube ends are stabilized by neighboring tubes. The tube ends are often found to be closed at the end of the process, but can be re-opened oxidatively and subsequently filled with gases and metals. As in the case of single wall tube synthesis, a large amount of side products are formed and separations are difficult.

Multiwalled tubes from a gas-phase catalyzed growth process are also available commercially at relatively low cost, but only under a restrictive nondisclosure agreement. Unlike the electric arc grown multiwalled tubes which are generally exceptionally linear, these tubes have defects that lead to bends and entanglements.

### Single Walled Nanotube Purification

Carbon nanotubes, despite the neat drawings often made about them, are rarely defect free and are often coated with a thin layer of amorphous material that is just discernible in TEM images. Such defects are the likely cause of decomposition in oxidative purification of single wall nanotubes, and this loss of material combined with the initial low yields of single wall tubes suggests that even the specialized use of such materials is many years in the future (an estimated cost for 1 pound of pure single walled nanotubes is \$3.5M).

## Goals and Scope

The goals of the LDRD were (1) to examine the chemistry of the nanotubes through both computations and experiment and (2) to investigate the electrical and mechanical properties that the nanotubes impart to composites. This project did not examine the physics or mechanical properties of nanotubes themselves as these areas were already under study at MIT and SRI. The study of the surface chemistry of the nanotubes is unique to SNL. A computational effort was then designed around these goals.

The scope of the project changed as difficulties in the purification of single walled nanotubes became apparent. The initial plans to examine both the surface chemistry of single walled nanotubes and the mechanical and electrical properties of their composites were changed after the first year. The second year focused instead on the electrical properties of multiwalled nanotube composites.

## **Results and Discussion**

The surface of a clean nanotube presents a "slippery" surface that is unlikely to provide an anchor for mechanical reinforcements. Our measurements of the modulus of composite materials containing impure nanotubes showed no significant improvement over base resins. In the case of typical carbon fiber composites, oxidative treatments are used to enhance fiber-matrix adhesion; however, such approaches are not available for single walled tubes whose structural integrity would be destroyed by such processes. Therefore computations were used to explore what type of surface functionalization might be possible on nanotubes.

### Computations

The computations yielded much insight into the chemistry of the nanotubes, which from these calculations, can be thought of as a highly-strained, polyaromatic, organic molecule. This strain energy is related to the curvature of the nanotube, thus the smallest diameter nanotubes, those with a diameter corresponding to  $C_{60}$  or  $C_{70}$  (about 1 nm), are the most reactive. However, calculations suggest that they are only reactive toward insertion of a carbene ( $R_2C:$ ) into the nanotube bonds, and not reactive to simple addition. This type of insertion is also calculated to be stable for insertion into the equatorial ring in  $C_{70}$ . The analogy is a valuable one, because it suggests that although the energetics of addition are favorable, the kinetics are unfavorable.

These results suggested that if pure samples of single wall nanotubes could be obtained from electric arc syntheses, only a carbene with virtually no other reactions available to it could be used. An excellent source for such carbenes are  $CCl_3SiF_3$  silanes from which  $:CCl_2$  can be obtained by heating to approximately  $140^\circ C$ . This carbene is formed in an essentially inert environment and would have essentially no other reaction pathways open to it other than addition to a nanotube. Furthermore, functionalization of the nanotube could be determined by x-ray fluorescence from chlorine.

### Nanotube Purification

Purification efforts -- despite continuing claims and carefully crafted TEM photographs -- do not yield pure materials. Our efforts included thermal treatments, air (oxygen) and aqueous ( $KMnO_4$  based) oxidations under many time/temperature combinations. These were all carried out with MER single wall materials. The problem that plagued SNL experiments were the same as those eventually acknowledged by many other researchers: that the roughly spherical glassy carbon that encapsulates most of the metal catalyst particles is less reactive toward oxidation than the nanotubes themselves and thus most, or sometimes all, of the nanotubes are destroyed

by such process long before the particulate carbon is consumed. Low yields (<10%) of still grossly impure materials were obtained. We were never able to carry out either mechanical tests on purified single walled tubes or attempt modification of the surface of the single walled tubes because of this purification issue.

Only after several months of purification experiments were the following conclusions reached: the project would either have to (a) fund the construction of an electric arc generation apparatus so that conditions could be determined that minimized the formation of particulate carbon, or (b) the project focus would have to move from single walled nanotubes to multiwalled nanotubes. And because the mechanical properties of the multiwalled tubes do not provide reinforcement (the inner and outer tubes are not necessarily the same length and are not chemically bound to one another, so the inner tubes simply pull out of an outer shell), the project focus was changed to electrical properties.

### Conductive Foams

Electrically conductive foams were first examined as a possible application for nanotubes. Such foams are of interest to other Sandia projects where a 3-dimensional, low density, electrical conductor would provide special advantages. Metal, or other particulate filled foaming resins, require high loadings to reach percolation and good conductivity. The unique high aspect ratios of the nanotubes provide high conductivity (single ohm centimeter values, depending on processing conditions) at loadings of only 0.5-1.0%. At these low loadings it is possible that the affects on the foaming behavior of the base resin would be minimized. A second advantage of using the nanotubes is also derived from the high aspect ratio of the nanotubes which leads to unusually high thixotropic behavior. In 1-2 week settling tests, absolutely no settling of the carbon nanotubes from the solvent was observed; after 4 months, only the slightest (perhaps 0.1%) amount of resin separated. This property reduces the need for other viscosity-modifying additives. Finally, these materials are opaque, which might yield certain additional advantages in some applications.

The foamable resin was filled with both carbon black (a conductive acetylene black) and vapor phase grown multiwalled nanotubes. The results of the electrical conductivity tests on both the base resin and the foamed resin are presented below.

### Hydrocarbon Resin Characterization

The samples were formed by a "boat" consisting of glass microscope slide base, two long walls formed by the thickness of other microscope slides and with copper electrodes at the ends. This formed a container of dimensions about 1.0 cm wide, 0.093 cm deep and 5.75 cm long. This allowed a small cross section and length long enough to easily measure volume resistivities over several orders of magnitude of current densities. The instruments used were Keithley 220 Constant Current Supply, Keithley 228A Constant Voltage or Current Supply, Keithley 617 Electrometer, Keithley 196 System DMM, Keithley 485 Picoammeter and Keithley 182 Sensitive Digital Voltmeter. The experiments were controlled by Labview on a MacIntosh computer. The current was ramped over several orders of magnitude while measuring the voltage drop across the sample. We calculated the volume resistivity versus the current density. The instruments could also be programmed to measure the volume resistivity versus time for a constant current density.

The first samples tested were number 1-1, 5% carbon black in hydrocarbon resin and number 1-2, 1% multiwalled nanotubes in a hydrocarbon resin (all samples were prepared by mixing in a ball mill at room temperature). Sample 1-1 had an original value of about  $7 \times 10^3$  ohm-cm at  $10^{-7}$  A/cm<sup>2</sup>. This value decreased to about  $1.3 \times 10^3$  at  $10^{-2}$  A/cm<sup>2</sup>. In driving the



higher currents through the sample, a permanent change in the sample occurred. The lower current density volume resistivity reduced to  $1.35 \times 10^3$  and little change if any at the higher current densities.

54°C..  $1 \times 10^4$  ohm-cm to  $6 \times 10^6$  A/cm<sup>2</sup> decreasing to  $1.7 \times 10^3$  ohm-cm at  $1.3 \times 10^3$  A/cm<sup>2</sup>.

21°C..  $2.8 \times 10^4$  ohm-cm to  $1 \times 10^6$  A/cm<sup>2</sup> decreasing to  $2 \times 10^3$  ohm-cm at  $1.3 \times 10^3$  A/cm<sup>2</sup>.

20°C..  $8.5 \times 10^3$  ohm-cm to  $7 \times 10^6$  A/cm<sup>2</sup> decreasing to  $1.5 \times 10^3$  ohm-cm at  $10^2$  A/cm<sup>2</sup>.

0.4°C..  $4.5 \times 10^3$  ohm-cm to  $7 \times 10^6$  A/cm<sup>2</sup> decreasing to  $1.5 \times 10^3$  ohm-cm at  $10^2$  A/cm<sup>2</sup>.

An increase in the temperature from 21°C to 54°C resulted in a 63% decrease in resistivity. A decrease in the temperature from 20°C to 0.4°C resulted in a decrease in the low current density resistivity by 20%, but there was little to no change above  $10^3$  A/cm<sup>2</sup>.

Sample 1-2 started at 480 ohm-cm from  $10^{-7}$  to  $10^{-3}$  A/cm<sup>2</sup> and then decreased to 320 ohm-cm at  $5 \times 10^{-2}$  A/cm<sup>2</sup>. The first scan caused permanent change to 390 ohm-cm at  $10^{-7}$  to  $10^{-3}$  A/cm<sup>2</sup> and reduced to 270 ohm-cm at  $5 \times 10^{-2}$  A/cm<sup>2</sup>.

52°C..  $8.7 \times 10^3$  ohm-cm to  $8 \times 10^5$  A/cm<sup>2</sup> decreasing to 220 ohm-cm at  $10^2$  A/cm<sup>2</sup>.

21°C.. 520 ohm-cm to  $8 \times 10^5$  A/cm<sup>2</sup> decreasing to 300 ohm-cm at  $10^2$  A/cm<sup>2</sup>.

21°C..  $1.2 \times 10^3$  ohm-cm to  $6 \times 10^5$  A/cm<sup>2</sup> decreasing to 220 ohm-cm at  $10^2$  A/cm<sup>2</sup>.

0.4°C.. 400 ohm-cm to  $3 \times 10^4$  A/cm<sup>2</sup> decreasing to 220 ohm-cm at  $10^2$  A/cm<sup>2</sup>.

An increase in the temperature from 21°C to 52°C resulted in a 21% increase in the resistivity. A drop in the temperature from 21°C to 0.4°C resulted in a 64% decrease in resistivities at low current densities, but no change above  $10^2$  A/cm<sup>2</sup>.

The second set of samples were 2-1, 20% carbon black in hydrocarbon resin and 2-2, 5% multiwalled nanotubes in a hydrocarbon resin. Sample 2-1 had a resistivity of 32.8 ohm-cm up to  $10^3$  A/cm<sup>2</sup> and then it dropped to 27.2 ohm-cm at  $2 \times 10^{-1}$  A/cm<sup>2</sup> and then rapidly increased at higher current densities. The temperature dependence was as follows:

50°C.. 35 ohm-cm to  $10^3$  A/cm<sup>2</sup> and drops to 25 ohm-cm at  $3 \times 10^{-1}$  A/cm<sup>2</sup>.

23°C.. 33 ohm-cm to  $10^2$  A/cm<sup>2</sup> and drops to 27 ohm-cm at  $3 \times 10^{-1}$  A/cm<sup>2</sup>.

0.3°C.. 24.7 ohm-cm to  $10^2$  A/cm<sup>2</sup> and drops to 22.7 ohm-cm at  $3 \times 10^{-1}$  A/cm<sup>2</sup>.

Sample 2-2 had a resistivity of 5.3 ohm-cm to  $10^2$  A/cm<sup>2</sup> and dropped to 3.15 ohm-cm at  $6 \times 10^{-1}$  A/cm<sup>2</sup>. The temperature dependence was as follows:

50°C.. 21.3 ohm-cm to  $10^3$  A/cm<sup>2</sup> and drops to 3 ohm-cm at  $6 \times 10^{-1}$  A/cm<sup>2</sup>.

23°C.. 5.3 ohm-cm to  $10^2$  A/cm<sup>2</sup> and drops to 3 ohm-cm at  $6 \times 10^{-1}$  A/cm<sup>2</sup>.

0.3°C.. 4 ohm-cm to  $3 \times 10^2$  A/cm<sup>2</sup> and drops to 3 ohm-cm at  $6 \times 10^{-1}$  A/cm<sup>2</sup>.

The third pair of samples were 3-1 20% carbon black in hydrocarbon resin and 3-2 5% multiwalled nanotubes in a hydrocarbon resin. This experiment yielded the resistivity as a function of time as it dried. At about 200 seconds, sample 3-1 had reduced to 12.6 ohm-cm and held steady to 90 minutes. At about 200 seconds, sample 3-2 had reduced to 1.87 ohm-cm and then started a slow increase to 3 ohm-cm at 90 minutes.

### Foam Preparation and Characterization

The final experiment was to form a sticky foam with 5% nanotubes and measure the resistivity versus time. The foam was produced inside a Lexan® box of dimensions 1.79 cm deep x 1.85 cm wide and 7.625 cm long. It had polished copper end plates for electrodes and copper wire through the boat for voltage pickoff. The wet resistivity started very high, several hundred megohm-cm. After 5 minutes, the resistivity had settled at  $6.8 \times 10^6$  ohm-cm and was not changing.

The low conductivity / high resistivity results were unexpected, and not explained until very recently. The process of expanding the foam works the materials in a manner such that the nanotubes tend to entangle and agglomerate. The agglomeration then leads to regions of low concentration of conductive material that act as high resistance or insulating volumes. Thus the overall material is non-conductive. There does not seem to be any means to foam such a material and retain the high electrical conductivity.

## **Continuing Research**

A CRADA was signed in order to exploit the electrical conductivity properties of these materials. A portion of the funds allocated to this project were used as Sandia matching contribution. At the conclusion of these studies a report containing CRADA-protected aspects of the project will be issued. Until final decisions regarding the intellectual property are made, please contact the authors for additional, potential commercially sensitive, information.

## **Conclusions**

The product of the electric arc process for nanotube growth contains either (1) single walled nanotubes that were neither separable from catalysts required for their formation nor from amorphous and graphitic carbons that were also present, or (2) multiwalled nanotubes, that may exist as isolated tubes or bundles, that are similarly not efficiently separable from large amounts of other carbons that are also produced. However, a satisfactory replacement for electrical applications are vapor-phase catalyst-grown multiwalled nanotubes which are available at low cost and high purity. Composites with these materials have low resistivity at unusually low loadings, but mechanical properties are not significantly affected. The viscosity of mixtures containing high loadings of such nanotubes is so high that adequate mixing is not currently possible.

Thus, structural composites of carbon nanotubes remain problematic. Even though calculations suggest that functionalization of single walled nanotubes should be possible, and that this may be a route to improved fiber-matrix binding, the inaccessibility and high cost of single walled nanotube fibers will continue to limit this line of inquiry. Multiwalled tubes show fiber "pull-out" from concentric cylinders; this limits their utility. Finally, the very real question is one of processing such materials into a useable form: our experience is that the viscosity of fiber loaded materials grows enormously at low loadings (and that dispersion is difficult and imperfect), such that only a few percent loading is possible, far less than might be desirable for structural applications.

The electrical properties of nanotube composites is interesting, potentially useful, and are the topic of a 100% funds-in CRADA in FY97. The low volume resistivities that can be obtained at ~1% loadings may be useful in both commercial and defense components.

## Abstracts of Published Manuscripts

The published work covers the work performed under case number 3501.360 (this project), and includes some work that was initiated under 3501.090 (fullerene research).

### "Ab initio computational study of selected $C_{60}H_6$ isomers"

Paul A. Cahill

Chem. Phys. Lett. **1996**, 254, 257-262.

The relative energies of eighteen isomers of  $C_{60}H_6$  have been determined through geometry-optimized ab initio calculations. HF/6-31G\* computations on the two lowest energy structures determined at the HF/3-21G level indicate that 1,2,4,11,15,30- $C_{60}H_6$  (**18**, with a  $C_{60}Br_6$ -like structure) lies only 0.4 kcal/mol higher in energy than 1,2,3,4,9,10- $C_{60}H_6$  (**4**). This remarkably close energy spacing was unexpected because, unlike in **4** in which addition occurs exclusively to 6,6 ring fusions,  $H_2$ -addition in **18** occurs both 1,2 to a 6,6 ring fusion and 1,4 across 6-rings so as to place two double bonds into 6,5 ring fusions.

### "Theoretical Studies of Derivatized Buckyballs and Buckytubes"

Paul A. Cahill and Celeste M. Rohlfiing

Tetrahedron **1996**, 52(14), 5247-5256.

Computational studies of patterns of addition to fullerenes have been extended (1) from  $C_{60}H_2$  and  $C_{60}H_4$  to  $C_{60}H_6$ , (2) from  $C_{60}H_2$  and  $C_{70}H_2$  to models of buckytubes- $H_2$ , and (3) from the products of the reaction of  $:CH_2$  with  $C_{60}$  to the corresponding products with buckytubes. A paradigm of multiple addition has appeared: as the number of  $H_2$  addends increases, 1,4 addition becomes competitive with 1,2-addition. A significant chemical difference between singly- (buckytube) and doubly- ( $C_{60}$ ) curved carbon is revealed by the energetics of  $H_2$  addition to the sidewalls vs. end-caps of a buckytube model. Remarkably,  $:CH_2$  insertion into either sidewalls or endcaps of buckytubes yields structures with similar heats of formation.

### "Semiempirical Study of Hydrogen Addition to Single-Walled Carbon Nanotubes"

Paul A. Cahill

Proceedings of the Electrochemical Society **1995**, 95-10, 1271-5.

Single-walled carbon nanotube models have been constructed by insertion of 10-carbon bracelets into  $C_{70}$  to form  $C_{90}$  and  $C_{120}$ . Semiempirical heats of vicinal hydrogenation along the sides of the tubes are  $\approx 40$  kcal/mol more endothermic (less stable) than addition to the endcaps. Based on the similarity of the endcaps to  $C_{60}$ , hydrogenation of nanotubes is estimated to be approximately thermoneutral; therefore, only relatively high energy dienes or other species are likely to yield stable addended products.

## Abstract of Accepted Manuscripts

“A Quantum Chemical Study of the Isomers of Bis(methano)fullerene, C<sub>60</sub>(CH<sub>2</sub>)<sub>2</sub>”

Celeste M. Rohlifing and P. A. Cahill

Molecular Physics, *in press*.

Building on our past success using quantum chemical predictions of relative isomer stabilities in guiding the synthesis of the hydrofullerenes C<sub>60</sub>H<sub>2</sub>, C<sub>60</sub>H<sub>4</sub>, and C<sub>70</sub>H<sub>2</sub>, we employ here semiempirical and ab initio methods to examine the eight isomers of bis(methano)fullerene, C<sub>60</sub>(CH<sub>2</sub>)<sub>2</sub>. The calculated lowest energy structure has two -CH<sub>2</sub>- units bridging adjacent junctions between 6-membered rings. Structural and energetic comparisons are made between the C<sub>60</sub>(CH<sub>2</sub>)<sub>2</sub> isomers and the analogous forms of C<sub>60</sub>H<sub>4</sub>. From a thermodynamic point of view, we predict that reactions of C<sub>60</sub> that produce C<sub>60</sub>(CH<sub>2</sub>)<sub>2</sub> (and C<sub>60</sub>(CR<sub>2</sub>)<sub>2</sub>) will yield mixtures of isomers, and not a few discrete products as found for the hydrofullerenes.

## Acknowledgments

Several staff, technicians and a post-doc contributed greatly to this project. Dr. Adra Baca, SMTS (now with AMP, Harrisburg, PA) with Ernie Correa, STA, performed all the mechanical testing. Dr. Todd Marquart, post-doctoral fellow (now with Motorola), exhaustively examined the nanotube purification. Paul Beeson, STA, performed all the electrical measurements on the samples that Brad Hance, STA, fabricated.

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