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Single Wall Carbon Nanotube Amplification: En Route to a Type-Specific Growth Mechanism

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Abstract: With the desire to mass produce any specific *n,m* type of single wall carbon nanotube (SWNT) from a small sample of the same material, we disclose here the preliminary work directed toward that goal. The ultimate protocol would involve taking a single *n,m*-type nanotube sample, cutting the nanotubes in that sample into many short nanotubes, using each of those short nanotubes as a template for growing much longer nanotubes of the same type, and then repeating the process. The result would be an amplification of the original tube type: a parent SWNT serving as the prolific progenitor of future identical SWNT types. As a proof-of-concept, we use here a short SWNT seed as a template for vapor liquid solid (VLS) amplification growth of an individual long SWNT. The original short SWNT seed was a polymer-wrapped SWNT, end-carboxylated, and further tethered with Fe salts at its ends. The Fe salts were to act as the growth catalysts upon subsequent reductive activation. Deposition of the short SWNT-Fe tipped species upon an oxide surface was followed by heating in air to consume the polymer wrappers, then reducing the Fe salts to Fe(0) under a H_2-rich atmosphere. During this heating, the Fe(0) can etch back into the short SWNT so that the short SWNT acts as a template for new growth to a long SWNT that occurs upon introduction of C_2H_4 as a carbon source. Analysis indicated that the templated VLS-grown long SWNT had the same diameter and surface orientation as the original short SWNT seed, although amplifying the original *n,m* type remains to be proven. This study could pave the way for an amplified growth process of SWNTs en route to any *n,m* tube type synthesis from a starting sample of pure nanotubes.

Introduction

Single wall carbon nanotubes (SWNTs) are regarded as ideal one-dimensional building blocks for electronic devices, electrical wiring, sensor arrays, and other nanoscale applications.[1] SWNTs can be best described according to their *n,m* types. The diameter and vector in which a graphene sheet is conceptually rolled to form a nanotube is defined by two integers, *n* and *m*, which define the nanotube type.[2] There are three cases for considering band structure from the *n* and *m* values. When $n - m = 0$, the SWNTs are metallic-like with a band gap of 0 eV and they are referred to as armchair nanotubes. When $|n - m| = 3q$, where q is a nonzero integer, the SWNTs are semimetallic-like, so-called mod-3 tubes, with bandgaps on the order of meVs. All other nanotubes are semiconductors with bandgaps varying between ca. 0.8 and 1.4 eV for HiPco-derived SWNTs. HiPco synthesized SWNTs produce about 80 different tube types, approximately two-thirds of those being semiconductors. All nanotube growth protocols form polydisperse (in *n* and *m*) nanotube batches, although some protocols are less polydisperse than others.[3] The wide variety of SWNT types that are coproduced in one batch of SWNTs, along with a lack of methods for preparatively well-separating those types, makes it difficult to construct devices that take advantage of the physical characteristics such as their band gap. The control of the *n,m* indices of synthesized SWNTs is a crucial factor for successful applications[4] utilizing their unique electric transport behaviors.[5] Although chemical and physical separation methods have been developed to sort SWNTs to some extent,[6] to date

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(1) For some recent examples, see: (a) Zhu, L. B.; Sun, Y. Y.; Hess, D. W.; Wong, C. P. *Nano Lett.* **2006**, *6*, 243. (b) Yuzvinsky, T. D.; Fennimore, A. M.; Kis, A.; Zettl, A. *Nanotechnology* **2006**, *17*, 434. (c) Stampfer, C.; Helbling, T.; Obergfell, D.; Schoberle, B.; Tripp, M. K.; Jungen, A.; Roth, S.; Bright, V. M.; Hierold, C. *Nano Lett.* **2006**, *6*, 233. (d) Merkoci, A. *Microchim. Acta* **2006**, *152*, 157. (e) Madden, J. D. W.; Barisci, J. N.; Anquetil, P. A.; Spinks, G. M.; Wallace, G. G.; Baughman, R. H.; Hunter, I. W. *Adv. Mater.* **2006**, *18*, 870. (f) Gruner, G. *Anal. Bioanal. Chem.* **2006**, *384*, 322.

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none have produced large quantities of *n,m*-controlled structures. The use of vapor liquid solid (VLS, a more accurate terminology in this case than chemical vapor deposition) growth methods is a promising protocol for the production of SWNTs with a narrow diameter distribution since the diameters of SWNTs correlate strongly with the sizes of metal catalyst particles,[7] but controlling the precise *n,m* type of SWNTs remains an unrealized synthetic challenge. While some of us encouragingly reported[8] the continued growth of original SWNT fibers, we did not show direct evidence of seeded growth from individually identifiable SWNTs since the growth of bundles of SWNTs, rather than individual SWNTs, was monitored.

Results and Discussion

The amplification process here was demonstrated microscopically on a single SWNT. Hence, this could lead the way to a methodology wherein, given a small pure *n,m* sample of SWNTs, one might use the nanotubes in that sample to serve as seeds and templates for generating more of that tube type. Thus the original small sample of pure *n,m* type SWNTs would be the progenitor of large quantities of the duplicated *n,m* type, in some ways similar to the polymerase chain reaction's amplification processes for DNA. The overall process is described in Scheme 1. Starting with HiPco SWNTs (**1**), they were baked in hot moist air and extensively washed in hydrochloric acid.[9] That cleaning process cracks any carbon shell around Fe, thereby exposing the Fe to dissolution in acid. The cutting process involved the previously described high-temperature fluorination to induce defects, hydrazine treatment to remove the fluorine, and final treatment with $H_2SO_4-H_2O_2$ to chemically cut, shorten, and end-oxidize the SWNTs.[9] This procedure further removes residual iron content in the HiPco generated SWNTs, and we have demonstrated that SWNTs treated by this protocol do not further grow upon C_2H_4 VLS growth exposure, with or without H_2 pretreatment.

The SWNTs were then polymer-wrapped with Pluronic F87 (MW = 7700, BASF), which is a triblock polymer of poly(ethylene glycol)/poly(propylene glycol)/poly(ethylene glycol), and subjected to ultracentrifugation to yield a decant that was >90% Pluronic-wrapped individual SWNTs (**3**), again carried out according to well-established protocols.[10] Throughout the process, ultrapure water was used (Barnstead, model D4751).

Polyethylenimine (PEI) (MW = 600, Sigma-Aldrich) was used to occlude $Fe(NO_3)_3$ and further form carboxylate/ammonium salt complexes with the carboxylated ends of the Pluronic-wrapped SWNTs (**3**). A PEI$-Fe(NO_3)_3$ stock aqueous solution made from 1 mg L^{-1} PEI and 4 mg L^{-1} $Fe(NO_3)_3$ was freshly prepared (the fresh preparation is important). One milliliter of the PEI$-Fe(NO_3)_3$ stock solution was mixed with 5 mL of aqueous Pluronic-wrapped SWNTs (**3**) (19 mg L^{-1}), and the mixture was gently stirred with a Teflon-coated magnetic stir bar overnight to yield the Pluronic-SWNT-PEI-$Fe(NO_3)_3$ (**4**) as depicted in Scheme 1.

A polished Si substrate covered with a native silicon oxide layer was inscribed with a locator recognition trench (Figure 1) using a diamond scribe; the end of the trench was used to image, by atomic force microscopy (AFM), the precisely same location of the substrate before and after VLS growth. The Pluronic-SWNT-PEI-$Fe(NO_3)_3$ (**4**) was deposited onto the substrate via dipping the silicon−silicon oxide substrate into the solution of **4**. Some of **4** was therefore located near the end of the recognition trench (Figure 1a). Before characterization, the sample was oxidized in air at 350 °C for 10 min to oxidatively remove the Pluronic and PEI portions from **4** while the $Fe(NO_3)_3$ converts to iron oxides (FeO) clustered about the SWNT's carboxylated ends to afford material as depicted by **5**. At this temperature in air, the carbon nanotube's framework is unreative.

The substrate supporting **5** was placed in a tube furnace and heated in H_2 (1 atm) for reductive docking treatment where the FeO is reduced to Fe(0) and can begin to etch into the SWNT, forming the SWNT-docked-Fe (**6**) seed. A majority of the SWNT seeds remained during docking at 500−750 °C for 5−30 min, but if longer docking times are used, few SWNTs remain since the Fe-catalyst particles, when no carbon feed gas is present, can efficiently etch into the tubes until there is no remaining SWNT carbon and this occurs rapidly at >750 °C. A particular 200 nm SWNT-docked-Fe (**6**) is apparent in Figure 1a (docked at 500 °C for 20 min). The height measurement result is shown in Figure 1b. The seed had a height of 0.73 nm, indicating an individual cut SWNT that was indeed no longer polymer-wrapped (the wrapping had been consumed during the initial thermal treatment described above; HiPco-derived SWNTs have diameters 0.6−1.4 nm[11]). The angle between its orientation and the left edge of the scribe mark is about 33°.

The VLS growth experiment was carried out in a normal electric furnace with a quartz tube chamber. Though our overall efficiency of amplification remains low (vide infra), our best conditions for clean and reproducible growth of **6** used C_2H_4 as the carbon growth source. The chamber containing the sample was purged with ultrapure argon gas at a flow rate of 1000 sccm for 1 h. The temperature of the sample was then ramped up to 750 °C in 20 min in the presence of 400 sccm of H_2 and 600 sccm of Ar. When the temperature reached 750 °C, C_2H_4 was introduced to initiate the growth of **7**. During the growth, the flow rates were kept at 100 sccm for C_2H_4, 400 sccm for H_2, and 500 sccm for Ar. Growth was continued for 5 min. After the growth, the flow of C_2H_4 and H_2 was halted, and the sample was cooled to room temperature under Ar. The cooled sample of the amplified SWNTs (**7**) was characterized using AFM (Figure 1).

A long and straight bidirectionally grown SWNT, 6.7 μm in length, imaged after the VLS amplification (Figure 1e), had the same orientation, relative to the inscribed recognition trench,

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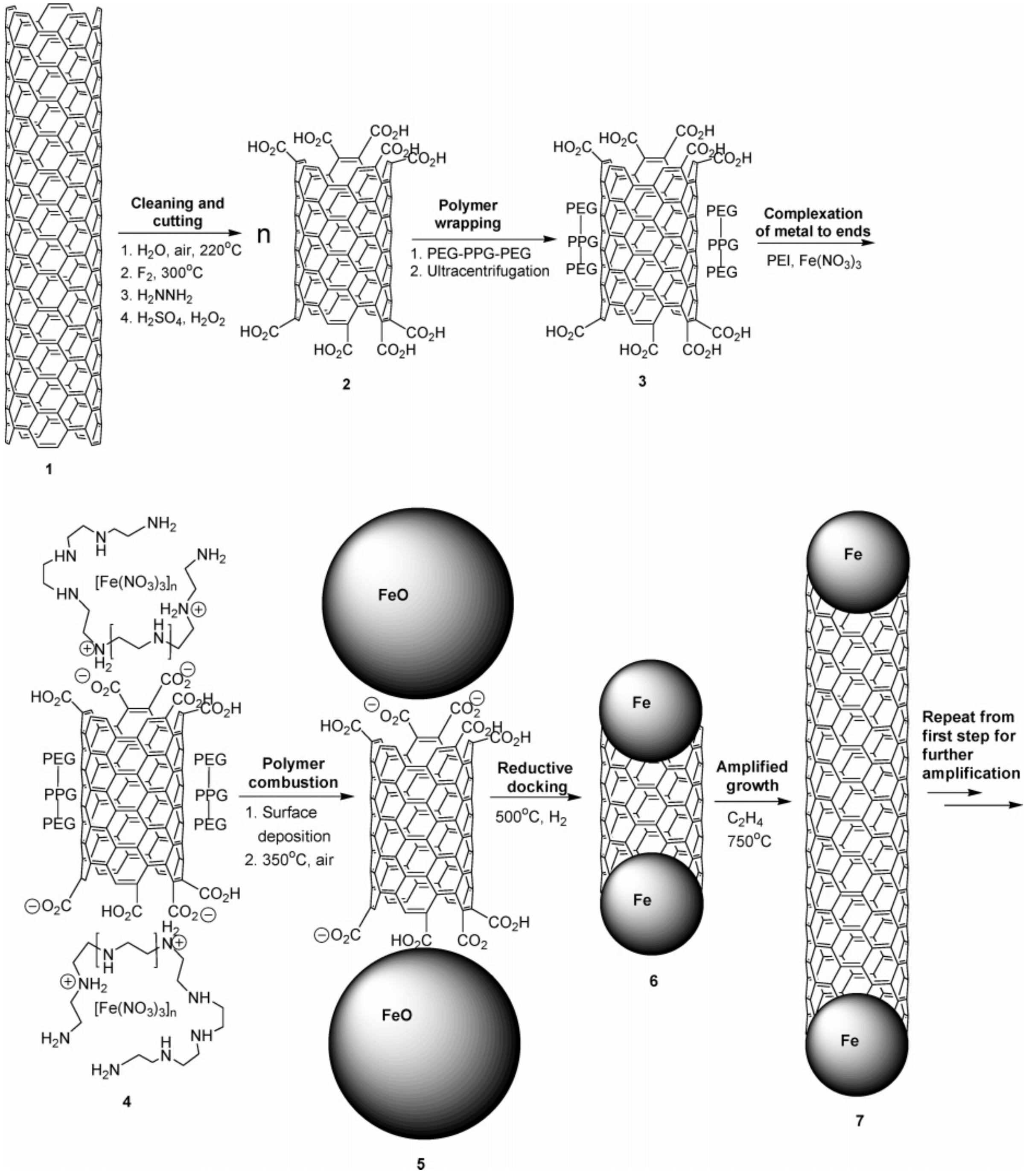
Scheme 1. A Depiction of the Amplification Growth Process of a Carbon Nanotube



HiPco-derived SWNTs (**1**) were cleaned and chemically cut to yield several ($n > 1$, and dependent on the original tube length and the cutting time) shorter fragments of nanotubes (**2**) that had oxidized ends that were predominantly carboxylic acids, but could also contain cyclohexenones. Polymer wrapping **2** with Pluronic F87 yields water-soluble nanotubes and an unbundled fraction, **3**, could be obtained by ultracentrifugation. Treating **3** with a mixture of PEI and $Fe(NO_3)_3$ affords SWNTs **4** that remain as individuals but have iron nitrate or oxide salts cocomplexed with the PEI that is acid−base associated with the nanotube ends. After depositing **4** on a silicon oxide surface, heating in air results in combustive loss of the Pluronic and PEI associating units, leaving iron oxides at the termini of the SWNTs as in **5**. Heating **5** under a H_2 atmosphere causes reduction of the iron oxides to Fe(0), and etching (reductive docking) of the Fe(0) into the SWNT, thereby generating a templated iron particle on the end of each SWNT as in **6**. When short **6** was then subjected to growth conditions in a C_2H_4 environment, the SWNT-templated Fe-nanoparticles amplified the templated nanotube structure forming long **7** with the same type characteristics as in **1**. Although not demonstrated here, the ultimate amplification process would subject **7** to the cycle again, thereby generating a repetitive method for carbon nanotube growth with the hope of selective *n,m* type amplification.

as that of the seed SWNT-docked-Fe (**7**) shown in Figure 1a. The height measurement for the amplified SWNT (**7**) in Figure 1d was 0.72 nm, identical, within error (± 3%), to the height of the original seed (**6**), suggesting a template growth of **6** to **7**. A large particle can be seen at the lower-left end of the grown tube (Figure 1e), indicative of carbonaceous growth around the original Fe-catalyst; it is this carbonaceous growth process that is believed to be the a source of termination in typical metal-catalyzed SWNT growth processes.[12] The fact that the seed

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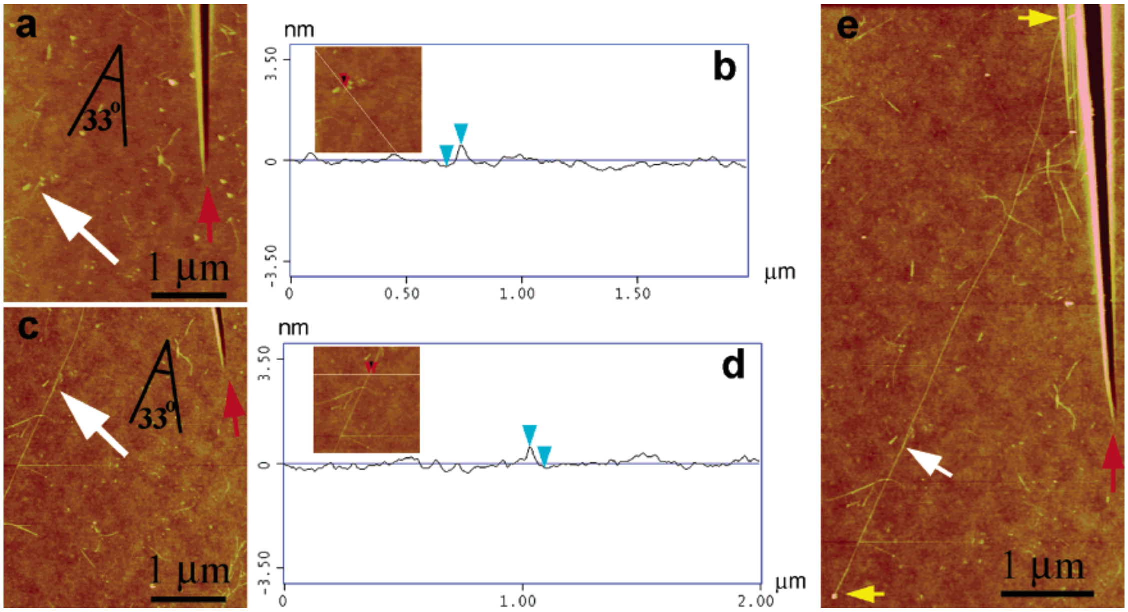

Figure 1. AFM images atop silicon−silicon oxide of a starting short SWNT and its amplified growth material. (a) The starting 200 nm long reductively docked seed-SWNT-Fe (**6**) (white arrow) is seen, while noting it position and angle relative to the locator inscription (red arrow). (b) A height measurement shows the seed-SWNT-Fe (**6**) to be 0.73 nm. (c) After exposure to the C_2H_4 growth conditions, the product amplified SWNT (**7**) (white arrow) had the same relative angle to the locator inscription (red arrow) as did **6**, and (d) nearly the identical height of 0.72 nm over several points measured along its entire length. (e) The entire length of the amplified nanotube **7** is 6.7 μm between the yellow arrows, with a white arrow noting the original SWNT seed location and a red arrow noting the locator inscription.

SWNT-docked-Fe (**6**) and the newly grown SWNT (**7**) had the same height and orientation relative to the inscribed trench mark indeed suggests that the SWNT in Figure 1d grew from the seed in Figure 1a.

Furthermore, that the newly grown nanotube segments could have been formed from residual Fe-catalyst remaining on the original nanotube from the HiPco growth can be ruled out by two factors. First, without reattachment of catalyst as described in Scheme 1, we have never observed continued growth after the acid/oxidation cleaning and cutting preparations described here. Second, and even more convincingly, the nanotube seeds described here grew bidirectionally. Any HiPco-derived nanotube only has Fe at one end. Therefore, in the unlikely event that Fe did remain at the end of a cut nanotube, it would have only been possible to be at one end and not both ends, as observed here. This ensures that the growth observed here was indeed seeded growth and not the result of residual Fe from the HiPco reactor.

A second amplified growth example [(using a solution of PEI−Fe$(NO_3)_3$ freshly prepared from 10 mg L^{-1} PEI and 4 mg L^{-1} Fe$(NO_3)_3$; 1 mL of this PEI−Fe$(NO_3)_3$ stock solution was mixed with 5 mL aqueous of Pluronic-wrapped SWNTs (**3**) (19 mg L^{-1}) and the mixture was gently stirred overnight to yield the Pluronic-SWNT-PEI-Fe$(NO_3)_3$ (**4**)] is shown in Figure 2. Reductive docking treatment was completed by annealing the sample in H_2 at 500 °C for 30 min, and the VLS growth was continued for 10 min under the same gas flow conditions as described above. Figure 2a shows the AFM images of a 480 nm SWNT-docked-Fe (**6**) recorded after the reductive docking (inscribed locator trench outside of the shown area, but a nearby pointed nanotube fragment can be used as a reference point). The height measurement (Figure 2b) shows that the seed is about 1.20 nm in diameter, suggesting that it is an individual SWNT,[11] not a bundle. As shown in Figure 2c, an amplified SWNT **7** formed at the same position as the original seed after the VLS growth. The diameter of the grown SWNT was 1.10 nm, as indicated in the height measurement result shown in Figure 2d, with a length of 7 μm (Figure 2e). Its orientation remained the same as that of the original seed. Figure 2d reveals that the tube passed over the inscribed recognition trench in the substrate during the growth. Therefore, this serves as a second example of seeded-SWNT amplified growth from an individual templated short SWNT.

A vast array of seed preparations from varying cutting protocols and functionalized (both covalent addends and noncovalent polymer wrappings) SWNTs, catalysts (based upon iron salts, iron−molybdenum clusters,[13] and mixed metals of Fe, Ni, and Co), surface deposition methods (spin coating, dipping, voltage driven depositions), surface substrates (silicon oxide, silicon hydride, magnesium oxide, highly ordered pyrolytic graphite, mica, and sapphire), and growth conditions including varying temperatures (500−950 °C), pressures, and growth gases (C_2H_4, CO, CH_4) were explored. Extreme care must be exercised, for example, CO is often contaminated with trace iron, which can catalyze new unseeded growth of SWNTs on a surface. Furthermore, traces of iron in the seeds solutions can promote new unseeded growth. Therefore careful image location and orientation must be analyzed and maintained between SWNT seeds and the VLS-grown products. Over the vast array of conditions studied, the best conditions are described here which minimized unseeded growth and maximized the seeded growth observed. Even with all this optimization, our best yields for seeded growth were only about 3%, meaning that most of the observed SWNTs on the surface never grew, and this can even be seen in Figures 1 and 2 where there are many nongrowing SWNTs. The catalyst concentration on the ends of

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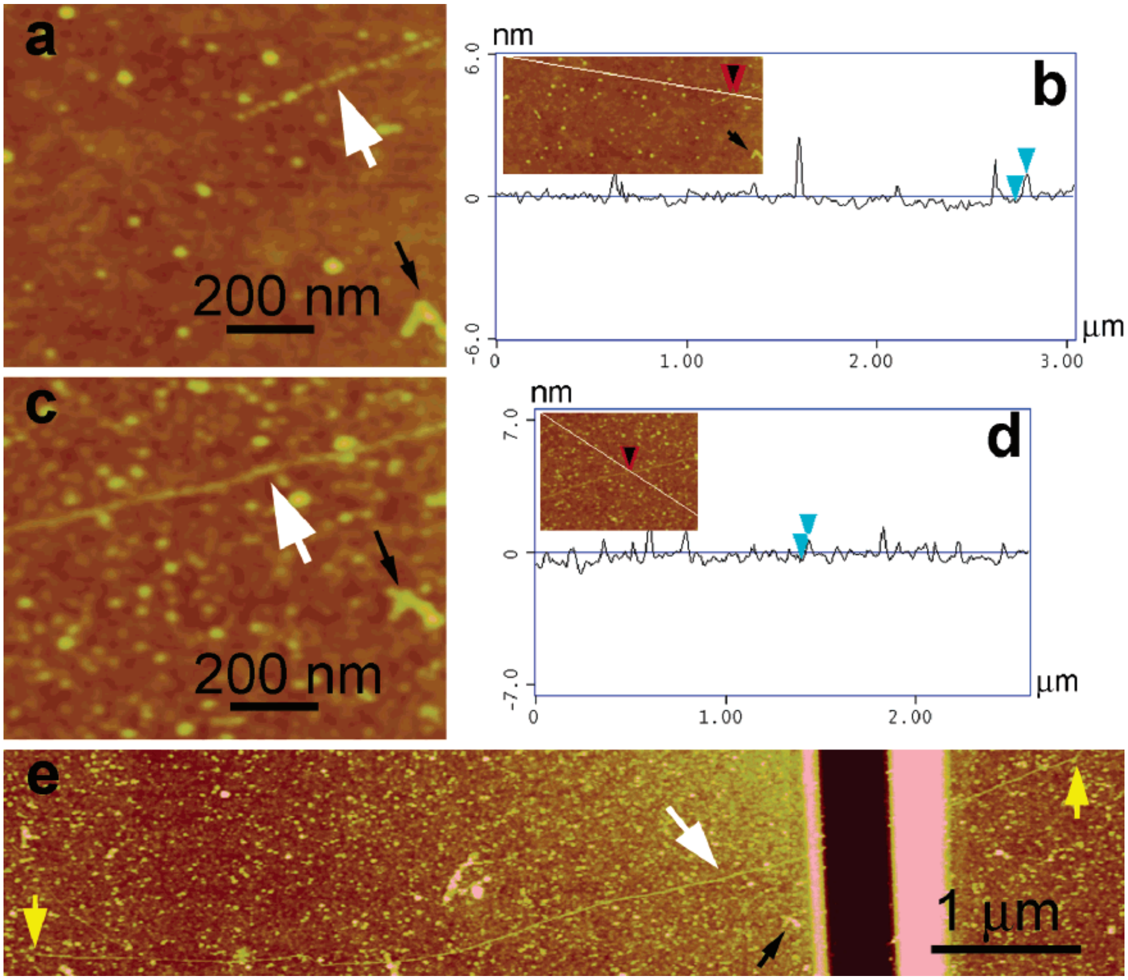

Figure 2. AFM images atop silicon−silicon oxide of a starting short SWNT and its amplified growth material. (a) The starting 480 nm long reductively docked seed-SWNT-Fe (**6**) (white arrow) is seen. Further, a pointed nanotube fragment can be used as a nearby reference point (black arrow). (b) A height measurement shows the seed-SWNT-Fe (**6**) to be 1.20 nm, again with a black arrow (inset) noting the pointed nanotube fragment reference. (c) After exposure to the C_2H_4 growth conditions, the product amplified SWNT (**7**) (white arrow) had (d) nearly the identical height of 1.10 nm over several points measured along its length. (e) The entire length of the amplified nanotube **7** can be seen to be 7 *µ*m between the yellow arrows with the original seed location (white arrow) and pointed nanotube fragment (black arrow) being noted.

the SWNTs was either too high or too low to see any amplification of most of the deposited SWNTs, and control of this factor is likely critical for an efficacious route to be developed. While precise catalyst concentration for reductive docking is not essential since even a large particle can result in the consumption of a SWNT, careful control of catalyst particle size is probably required in this template growth method since carbon overcoating of the metal or tube pinch-off could be common growth termination sources.[12] The preparations of active SWNT seeds are presently low-yielding and the docked products are impossible to characterized when it comes to assessing the number of metal atoms per tube end. We invested a great deal of effort to use more well-defined metal clusters for docking to the ends of SWNTs, namely, the well-defined Fe−Mo cluster $[H_xPMo_{12}O_{40}\subset H_4Mo_{72}Fe_{30}(O_2CMe)_{15}O_{254}(H_2O)_{98}]$, which has been used for surface growth of unseeded SWNTs,[13] but again, the results were inferior to those reported here. Therefore, the amplification result here, albeit low-yielding, is unprecedented. Furthermore, to be ultimately successful in generating specific tube types from a given sample, the process requires fractionation of the SWNT types to be done once, on a small scale, followed by efficient cutting and high fidelity amplification based on seeded growth, for the requisite replicating process. Therefore, the work described here is merely a proof-of-concept in the overall goal of large-scale amplification. Moreover, our analysis of nanotube-type fidelity is based solely on height; therefore, further assessment will be critical using microscopic Raman analysis to verify retention of *n*,*m* type between the original and new segments.

Summary

In summary, the growth of SWNTs from individual short SWNT seeds has been demonstrated. The process involved the sequence of templated VLS growth of individual SWNTs. The original SWNT systems were short polymer-wrapped individual SWNTs, end-carboxylated, and further tethered with Fe salts. Deposition of the SWNT-Fe salts upon an oxide surface was followed by heating to consume the functional polymer wrappers while reducing the Fe salts to Fe(0) under a H_2-rich atmosphere. During this heating, the Fe(0) can etch into the SWNT, thereby reductively docking into the SWNT so that the SWNT acts as a template for elongation growth that occurs upon introduction of a carbon source. Analysis indicated that each VLS-grown SWNT had the same height and surface orientation as the original SWNT seed, thereby suggesting amplification of the original nanotube. The large increase in length, to several micrometers, was achieved during 5–10 min growth processes. This study establishes a method for an amplified growth process of SWNTs with the hope of duplicating any desired *n*,*m* tube: a process that will be required for many electronics and optoelectronics applications.

Acknowledgment. We thank B. Chen, J. Hudson, L. McJilton, S. Pheasant, Z. Gu, R. Anderson, D. Ogrin, H. Peng, C. Kittrell, and E. Billups for assistance. This work was supported by the Defense Advanced Research Projects Agency, the National Aeronautics and Space Administration, and the Robert A. Welch Foundation under Contracts N00014-04-1-0765, NNJ05HB73C, and C-0689, respectively.

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