Chapter 6

Self-assembled Semiconducting Single-Walled Carbon Nanotubes by Polyfluorene Derivatives Containing Thiol Groups

This chapter demonstrates the selection of s-SWNTs by employing polyfluorenes with side chains containing thiol groups aiming for self-deposition of SWNT onto the substrate. We show that the inclusion of thiol groups to the polymer disrupts the s-SWNTs selection, with the presence of metallic tubes in the dispersion. The selectivity can be recovered either by adjusting the number of thiol groups in the polymer, or by fine tuning the polymer to SWNT ratio. We demonstrate that the polymer containing 2.5% thiol group gives the best s-SWNT purity, as confirmed by photoluminescence spectroscopy measurements. Field-effect transistors fabricated by direct self-assembly of the SWNTs/thiolated-polyfluorenes on gold patterned substrates show superior performances (mobility up to 16 cm²/V·s) with 3 orders of magnitude higher on-current compared to transistors fabricated with SWNTs wrapped with the same polymer without thiol functionalization.

6.1 Introduction

Single-walled carbon nanotubes (SWNT) are one of the most prospective candidates to succeed silicon for future electronic devices “beyond Moore’s law”. The application of nanotubes as active material for electronic devices started in the early 2000s, but interest in them initially declined due to difficulties in the separation of semiconducting species from the metallic ones. Recently, the possibility to purify s-SWNTs has shown very remarkable progress, especially the polymer wrapping technique, which is the subject of this thesis. It has shown great potential owing to the ability to achieve near 99.9% purity. \[1\] After successful s-SWNT separation, current research concentrates on demonstrating the usefulness of carbon nanotubes by fabricating highly performing field-effect transistors,\[^{2,3}\] light-emitting devices,\[^{4,5}\] logic gates,\[^{6}\] sensors,\[^{7}\] and photovoltaics devices.\[^{8,9}\]

As already mentioned in previous chapters, it is very important to have high concentration and high purity s-SWNTs in order to obtain outstanding device performance. To achieve these requirements, optimization of the s-SWNT selection process parameters, such as the nature of the solvent (chapter 2), the sample preparation and sonication temperature (chapter 3), or the use of different polymer structures (chapter 4 and 5) have been intensely investigated. Another important point to boost the applicability of s-SWNT in electronic devices is to achieve a good control of the SWNT placement in specific positions, which will open the way to mass production of SWNT electronics. Carbon nanotubes are anisotropic nanomaterials, it is therefore fundamental to have them deposited in specific direction and position, i.e. perpendicular to the source-drain electrodes in transistor with very high precision and reproducibility. In order to obtain a controllable deposition, two distinct approaches are generally used: self-assembly into desirable structures, and special techniques to deposit an aligned nanotube film.\[^{10}\]

Aligned nanotube networks perpendicular to the source-drain electrodes in the SWNT-FET have been shown to demonstrate superior performance compared to random network films.\[^{2,11–14}\] Here, the presence of fewer percolation channels is one of the main reasons for the higher performances. Several works have already been reported to achieve fully or partially aligned SWNT networks by blade coating\[^{2}\] or by the Langmuir-Blodgett technique.\[^{12}\] Different approaches have been developed to obtain a dense and aligned carbon nanotube network by self-deposition of SWNTs to specific areas, contact electrodes or substrates, such as solvent evaporation-assisted
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self-assembly,[12] dielectrophoresis,[14] or by selective placement via direct self-assembled molecules such as DNA pairs,[15] and functionalized groups that have a specific interaction with the substrates/electrodes.[16]

In this chapter, we report a mechanism of directly assembling s-SWNTs to gold electrodes, by wrapping SWNTs by polyfluorene derivatives decorated with thiol functional groups that can bond covalently to the gold surface. The ideal schematic of the self-assembled SWNTs on the gold substrate is illustrated in Figure 6.1. We found that inserting a thiol group decreases the s-SWNT selection due to the lone electron pairs on sulfur atoms that can interact with the highly polarizable metallic tubes. A set of polymers with different thiol-group content were synthesized to study the effect of the thiols on the s-SWNT selection. We concluded that polyfluorene decorated with 2.5% thiols at the side-chains gives the highest purity of s-SWNT. These high purity semiconducting tubes are further employed for self-deposition onto silicon substrates with pre-patterned gold electrodes. The devices fabricated by s-SWNTs wrapped with thiol-containing polymer show ambipolar properties with 3 orders of magnitude higher on-current compared to the reference devices fabricated with SWNTs selected with a polymer without thiol functionalization. AFM images demonstrated high-density network of carbon nanotubes for the sample obtained with the thiol functionalized polymer, indicating the effective self-assembly in the used substrate.

![Figure 6.1 Schematic thiol attachment to the gold substrate in SWNT-FET devices.](image-url)
6.2 s-SWNT selection by polyfluorene derivatives containing thiol functional groups

In recent years, polyfluorene derivatives have been widely investigated for semiconducting SWNT selection due to their capability to interact preferentially with nearly armchair carbon nanotubes.\[^{17}\] Several approaches have been applied to modify the polyfluorene to obtain higher or better s-SWNTs selection, such as modifying the alkyl side-chains\[^{18}\] or the polymer backbones\[^{19}\]. Our group has reported a different approach of polyfluorene modification, which had the aim of promoting the self-assembly of semiconducting carbon nanotubes onto gold substrates. This was achieved by synthesizing polyfluorene-DNA block copolymers, where the DNA pairs were used as anchoring molecules for the self-deposition.\[^{15}\] However, this method shows some major drawbacks, which are the difficulties and high production cost to make sequentially controlled DNA pairs and to synthesize the block-copolymers. In order to obtain a cheaper way to perform direct self-assembly of the nanotubes to gold electrodes, different self-assembled molecules need to be considered, for example thiol groups, that have specific interaction with gold.\[^{20}\] Therefore, we adopted polyfluorene derivatives decorated with thiol functional groups on their side-chains or at the end of the polymer chain.

Figure 6.2a shows the absorption spectra of HiPCO SWNTs selected by polyfluorene derivatives containing different percentage of thiol groups. Among all, the reference polymer containing no thiol group, Poly\{\text{(9,9-didodecylfluorene-2,7-diyl)\_alt\-(9,9-dihexylfluorene-2,7-diyl)}}\ (PF12-F6), shows the highest concentration of s-SWNTs. Introduction of the thiol functional group to the polymer disrupts the SWNT/polymer interaction, resulting in lower absorbance of the s-SWNTs dispersion. Furthermore, when a high concentration of thiol groups (50%) is attached to the polymer side-chains (PF12-F6Sac), a lower selectivity for s-SWNTs is obtained, as indicated by the high background absorption, which is due to the metallic SWNT plasmon resonances, and by the broader absorption peaks due to the overlap between individual nanotube peaks. Moreover, a small percentage of metallic SWNTs is also observed in this dispersion as indicated by several peaks in the range between 500 and 650 nm. The presence of the metallic tubes in this high thiol concentration polymer/SWNT dispersion is analogous to previous reports\[^{21}\] where metallic SWNTs are selected by polymers containing high concentration of nitrogen atoms, due to the electron rich nature (lone pair) of the nitrogen atoms. The electron lone pairs on the
thiols (sulfur atoms) are similar to those in nitrogen atoms, which can interact with highly polarizable SWNT species, the metallic ones in this case.

To reduce the metallic content in this dispersion, several different approaches can be utilized. The first is to reduce the number of thiol groups in the polyfluorene chain.

![Absorbance of s-SWNT dispersion with polyfluorene derivatives containing 0% (black), 2.5% (red), 50% (green) of thiol groups at their side-chains, and containing thiol groups as end-chains (blue). Dispersions are prepared with the same initial ratio. (b) Absorbance similar to sample (a) after optimization to remove the metallic SWNT, and after enrichment. Insets: Structures of the polymers used for the dispersion of the SWNTs.](image-url)
As seen clearly in Figure 6.2a, reducing the number thiol groups to 2.5% (PF12-F6SAC2.5%) reestablishes the selectivity for s-SWNTs. When only 2 thiol functional groups at the end of polymer are present (PF12-α,ωSAC), the purity of s-SWNTs in the supernatant solution is increased, demonstrated by lower background absorption.

Another route to reduce the metallic content is through fine tuning of the polymer:SWNT ratio. It has been reported that by increasing the SWNT to polymer ratio, a lower concentration but higher purity s-SWNT dispersion can be realized.[22] The absorption spectra of enriched SWNT dispersions with these polymers after optimization (see experimental details) and free polymer removal are displayed in Figure 6.2b. The assignment of s-SWNT chiralities is displayed in the figure, according to the previously reported formula (equation 2.1, page 34). All polymers select the same species of SWNTs, indicating that the placement of the thiol functional groups at the side-chains does not affect the selection of s-SWNTs species. Interestingly, the inclusion of thiol at the end of polymer backbones modifies the distribution of selected s-SWNTs, i.e. this polymer selects smaller diameter tubes. The s-SWNT species selected by PF12-α,ωSAC are more similar to the ones selected by Poly{(9,9-dioctylfluorene-2,7-diyl) (PF8). We hypothesized that the different selection can originate from (i) different steric hindrance induced by the thiol groups in the side chain and (ii) smaller molecular weight of the polymer.

To verify the quality of our s-SWNT dispersions, photoluminescence measurements were performed on the enriched samples where the final concentration was adjusted to similar values (samples of Figure 6.2b). The steady state PL measurements are shown in the Figure 6.3a, showing that the reference sample has the highest PL intensity, while increasing the number of thiol groups reduces the PL intensity. The lowering of the steady state PL intensity can be attributed to the presence of bundles, which may also contain metallic nanotubes. Time-resolved spectroscopy measurements can be used to further elucidate the origin of this phenomenon. The exciton decay dynamics of (7,5) tubes are shown in Figure 6.3b. In general, the long decay component of the exciton lifetime of the individualized s-SWNTs is in the range of 20-30 ps, which is comparable to the lifetimes previously reported for highly individualized polyfluorene-wrapped s-SWNTs,[18,23,24] demonstrating the absence of metallic carbon nanotubes in the dispersions. It has been reported that when metallic SWNTs are present in the solutions, the exciton lifetime of s-SWNTs will reduce by one order of magnitude.[25] The nanotubes selected by PF12-F6SAC show the shortest lifetime, in agreement with
the lowest steady state PL, confirming the highest number of pathways for exciton relaxation due to the formation of s-SWNTs small bundles.

Figure 6.3 (a) Steady state photoluminescence of the s-SWNT solutions, dispersed by polyfluorene with different concentration of thiol groups (b) Time-resolved measurement of (7,5) tubes (1050 nm) dispersed by polyfluorene with different concentration of thiol groups.

6.3 Self-assembly of SWNTs onto substrates with pre-patterned gold electrodes

The selective deposition of s-SWNTs by specific techniques such as solvent evaporation-assisted self-assembly, dielectrophoresis, etc. have been challenging, due to the lack of selective deposition and the necessity of complex methods such as pre-patterning of the substrate with chemical species to attract SWNTs. One possible approach for more controllable deposition of SWNTs is the application of directed self-assembled molecules. The self-assembly technique has the advantages of being a simple process and the attachment to the substrate/electrode is by covalent bonding, which is therefore highly robust, thermally stable, and chemically resistant to
a variety of reagents. Directed self-assembly for SWNTs has been reported using single strand DNA,[15] covalent functionalized groups, or ionic functionalized groups.[16]

Self-assembled monolayers of molecules with thiol functional groups have been studied for a very long time, an ubiquitous example is the chemisorption of thiols on gold by the formation of the S-Au bond at the gold surface.[27] Other metals that show specific bonding with thiols are silver, copper, and palladium. For real-world applications of self-assembly, gold and palladium are the most attractive metal electrodes due to high chemical stability, low toxicity, and good compatibility for CMOS technology.[20] To demonstrate the usefulness of thiol groups in the conjugated polymers used to wrap the SWNTs, and to self-assemble them, we fabricated SWNT field-effect transistors using gold as source and drain electrodes.

The sorted s-SWNTs were deposited by soaking the silicon substrate with the pre-patterned gold in the enriched dispersion for 1 hour. Figure 6.4a and c show the comparison of the output curves of the SWNT-FETs fabricated by SWNTs selected by PF12-F6SAC2.5% and the reference polymer (PF12-F6), respectively. Both devices show ambipolar transport with almost symmetric electron and hole current, demonstrating the well-balanced electron and hole populations in the channel. The devices fabricated from SWNTs wrapped by thiolated polymer show 3 orders of magnitude higher on-current compared to that of the reference devices, demonstrating better SWNT attachment to the electrode/substrate during the immersion time. Compared to the reference devices, these devices also show better carrier injection as indicated by the shape of the output characteristics at low source-drain voltage.

A noticeable reduction of currents at high gate and source-drain voltages is observed from the output curves of the SWNT-PF12-F6SAC2.5% sample, a similar effect has been reported for many ambipolar devices.[28] In such devices, the holes and electrons can be injected simultaneously from both electrodes. At low source-drain voltage, only one type of charge carrier can be present in the active layers for example holes (electrons) for negative (positive) gate bias. The carriers with opposite sign are blocked due to a high Schottky Barrier at the source/drain electrodes. When the source-drain voltage is further increased, the opposite carrier starts to be injected into the active layer due to the lowering of the barrier. At this high voltage, both carriers exist in the active layer, and can therefore recombine, resulting in the reduction of the current. Since carbon nanotubes are 1D- direct band gap materials with strong exciton binding energy, the electron and hole recombination might result in light emission that has been demonstrated by different authors.[4,5,12]
The transfer characteristics of corresponding SWNT-FETs fabricated by SWNTs selected by PF12-F6SAc2.5% and PF12-F6 are presented in Figure 6.4b and d. Both devices show on/off ratio around $10^4$ and $10^5$ at $V_{DS} = \pm 5$ V for electrons and holes, respectively. Also the increase of the off-current by increasing source-drain voltage is a typical characteristic of ambipolar transistors. Since both carriers can be injected into the channel simultaneously, the devices cannot be completely turned off at high drain voltages. The charge carrier mobilities were extracted from the $I_D$-$V_G$ characteristics using equation 1.3 (page 17) in the linear region at $V_{DS} = \pm 5$ V. For the devices fabricated with PF12-F6SAc2.5%-SWNTs, we obtained effective mobilities of 9.15 cm$^2$/V·s and 15.97 cm$^2$/V·s for holes and electrons, respectively. The reference devices show mobilities of 0.05 cm$^2$/V·s and 0.07 cm$^2$/V·s for holes and electrons, respectively.

The mobility values obtained with the self-assembled SWNTs are extremely high especially considering their perfect ambipolarity, and the fact that small diameter carbon nanotubes and a very simple device structure (and dielectric) are used. For comparison, these mobilities are close to those obtained by our group with ambipolar transistors (14 and 16 cm$^2$/V·s) fabricated using large diameter nanotubes (1.5nm diameter) and ion gel as gate dielectric.\[18\]
Selecting s-SWNTs by polymer wrapping: Mechanism and Performances

![Graphs](image)

Figure 6.5 Comparison of transfer curve before (solid line) and after (dashed line) sonication for (a) PF12-F6SAc2.5%-SWNTs and (b) PF12-F6-SWNTs.

To prove that the high performance of the field effect transistors fabricated with PF12-F6SAc2.5%-SWNTs is due to the strong attachment of the SWNTs to the substrate, we performed a mild sonication of the devices in toluene after electrical characterization. Interestingly, after sonication and re-annealing to remove the solvent, the electrical properties of the samples fabricated by thiol self-assembly show only a decrease of the on-current by 1 order of magnitude (see Figure 6.5a), while the reference sample shows a reduction of the on-current by more than 2 orders of magnitude from the already much lower starting value (Figure 6.5b).

Atomic Force Microscopy micrographs of the SWNT networks, obtained with PF12-F6SAc2.5%-SWNTs and PF12-F6-SWNTs dispersions, recording the transistor active channel (bottom regions) and the gold electrodes (top regions) are displayed in Figure 6.6b and c, respectively. In Figure 6.6b, we clearly observe a homogeneously covered network of highly individualized carbon nanotubes, both on the substrate and...
on the gold electrodes, having a diameter of ~3 nm (measured from the cross sections). On the other hand, the reference sample (Figure 6.6c) shows sparse coverage of SWNTs with the presence of many bundles in the channel, and even more sparse coverage on the gold electrodes. The thiolated sample also shows a larger number of SWNTs at the interface with the gold electrode, making a better contact, and therefore explaining the better injection and lower Schottky barrier of these samples. Another interesting observation is that the sample containing thiols on the side-chains of the polymer shows significant amounts of small dots, while the small dots are absent in the reference sample. The small dots in the AFM figure, which have average height of 3 to 7 nm, most likely come from the aggregation of the alkyl side-chains of the polymers due to strong polarization of thiol groups, or from S-S bond formation.

Furthermore, we performed AFM measurements of the thiolated SWNT sample drop casted on an ultra-flat MICA surface, where the recorded AFM pictures confirms the presence of the same small dots observed on the Si/SiO₂ substrate (Figure 6.4a), which clarifies that their presence is not linked to the sample preparation method or the specific surface nature (MICA surface is negatively charged).

Observed with AFM, the homogeneous SWNT network on the Si/SiO₂ and gold electrodes proves our initial hypothesis that thiol functionalization could play an important role in the fabrication of homogeneous SWNT networks and also aid in efficient contact formation with the gold electrodes by the application of a simple dipping technique, which is extremely promising for large area fabrication technology.

6.4 Conclusion

In conclusion, we demonstrated the selection of semiconducting SWNTs by polyfluorene derivatives decorated with thiol functional groups, either at the alkyl side-chains or at the end of the polymers. Due to the polarized electron lone pairs, the thiol group impairs the selection of semiconducting tubes. Nevertheless, the metallic content can be minimized by adjusting the concentration of the thiol functional groups, or by using different SWNT to polymer ratio. The high purity of individualized s-SWNTs was further proved by photoluminescence spectroscopy, showing the long exciton lifetime for all samples after optimization of the selection procedure. The self-assembly of the SWNT/thiolated-polyfluorene is utilized for SWNT-FETs fabrication, showing superior performance, indicated by 3 orders of magnitude higher on-current with respect to the reference sample fabricated with SWNTs selected with the non-thiolated polymer. This superior performance is attributed to higher coverage of
SWNTs and better contact between the SWNT networks and the gold electrodes, as demonstrated by AFM imaging.

6.5 Methods

**Polymer synthesis.** The synthesis of $\alpha, \omega$-Bis(4,4-phenyl-1,1-thioacetate)-poly(9,9-didodecylfluorene-2,7-diyl) (PF$_{12}$-$\alpha, \omega$SAc) was carried out in a 100 mL Schlenk-tube equipped with a magnetic stirring bar under inert atmosphere. 615 mg (3.94 mmol) 2,2'-Bipyridyl und 1166 mg (4.24 mmol) bis(1,5-cyclooctadiene)nickel(0) were added to this tube. Under exclusion of light, 45 mL toluene, 15 mL DMF and 0.48 mL (3.94 mmol) 1,5-cyclooctadiene were added, stirred and slightly heated. 1000 mg of the monomer (1.54 mmol) 2,7-bibromo-9,9-didodecylfluorene and 35 mg of the endcapper (0.151 mmol, 10 mol%) 4-bromophenylthioacetate were added in a second tube, diluted in 15 mL toluene under inert atmosphere, and added to the warm reaction mixture. The reaction was stirred for four days at 80 °C.

The reaction was terminated by quenching with 10 mL 4M hydrochloric acid. Afterwards, chloroform was added to the mixture and the solution was subsequently washed with 2M hydrochloric acid, saturated sodium hydrogen carbonate solution, EDTA, and brine. The resulting polymer solution was concentrated and precipitated from acidic cold methanol. As a further purification step, a Soxhlet extraction using methanol, acetone, ethyl acetate, and chloroform was conducted. The isolated chloroform solution was stirred with aqueous sodium diethyldithiocarbamate solution to remove catalyst residues. After separation of both phases and washing of the polymer solution with water, another precipitation from cold methanol was performed, resulting in a pale-yellow polymer yielding 59 % (462 mg, 0.92 mmol).

Poly{(9,9-didodecylfluorene-2,7-diyl)-alt-}[9,9-bis[6-(S,S’- \text{thioacetate})hexyl]fluorene-2,7-diyl]} (PF$_{12}$-F$_6$SAc) was prepared by adding 503 mg potassium thioacetate to diluted solution of 450 mg of poly{(9,9-didodecylfluorene-2,7-diyl)-alt-}[9,9-bis(6-bromohexyl)fluorene-2,7-diyl)]. After addition, the polymer solution was stirred at 70 °C and after twelve hours, the solvent was removed, chloroform added, and the polymer precipitated from cold methanol two times. The filtrated yellowish polymer was dried under high vacuum yielding 77 % (333 mg, 0.34 mmol).

Poly{[(9,9-didodecylfluorene-2,7-diyl)-stat-}[9,9-bis[6-(S,S’-\text{thioacetate})hexyl]fluorene-2,7-diyl]} (PF$_{12}$-F$_6$SAc$_{2.5}$%) was prepared by adding...
1000 mg (1.51 mmol) 2,7-dibromo-9,9-didodecylfluorene and 68 mg (0.11 mmol) 2,7-dibromo-9,9-bis(6-(S,S'-thioacetate)hexyl)fluorene to a Schlenk-tube. Under inert atmosphere, 615 mg (3.94 mmol) 2,2'-bipyridyl and 1166 mg (4.24 mmol) bis(1,5-cyclooctadiene)nickel(0) were added and diluted in 15 mL anhydrous THF. Furthermore, 0.45 mL (3.94 mmol) 1,5-cyclooctadiene were added and the resulting mixture was heated in a microwave reactor at 120 °C for ten minutes. Afterwards, 0.1 mL p-bromobenzene (0.95 mmol) were added and the reaction mixture reacted for five more minutes at 120 °C. The workup was done analogous to the of PF12-F6SAc yielding 705 mg (1.07 mmol, 87%) pale-yellow polymer.

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<td>1.55</td>
</tr>
<tr>
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<td>55,000</td>
<td>1.58</td>
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<tr>
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<td>114,000</td>
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<td>1.82</td>
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Table 6.1 Molecular weight data of the polymers

**Dispersion of semiconducting SWNT.** The SWNT dispersion are prepared following the previously reported methods (chapter 3). Specifically, a mixture of SWNT:polymer with 3:12 weight ratio in 10 mL of toluene was sonicated in a cup horn bath sonicator (Misonix 3000, output power 65 W) and then ultracentrifugated at 30,000 rpm / 110,000 g (Beckman Coulter Optima XE-90; rotor: SW55Ti). The supernatant is taken and used for absorption measurement. Different SWNT to polymer ratios were adopted to obtain the highest s-SWNTs purity for the 50% thiolated-polyfluorene, with the optimal ratio being the 6:6 weight ratio. A second step ultracentrifugation was performed at 55,000 rpm / 367,000 g to remove the excess (free) polymer chains, and to increase the s-SWNT concentration.

**Optical absorption spectroscopy.** Absorption spectra were recorded in a wavelength range from 300 to 1800 nm in 2 mm path length quartz cuvette using an UV-Vis-NIR spectrophotometer (Shimadzu UV-3600).

**Photoluminescence measurements.** Steady state and time resolved photoluminescence spectroscopy was performed by exciting the s-SWNT solutions with a mode-lock Ti:Sapphire laser at wavelength of 800 nm. The duration of the pulses is 150 fs and the repetition frequency ~76 MHz. Steady state photoluminescence spectra were recorded with an InGaAs photodetector array from Andor while the time resolved
measurements were recorded with a streak camera with an NIR-sensitive photocathode from Hamamatsu Photonics working in synchroscan mode. All measurements were performed in transmission mode with the sample at room temperature.

**Device fabrication and characterization.** A highly doped silicon substrate with 230 nm of SiO₂ dielectric layer and patterned electrodes made with 10 nm of Ti and 30 nm of Au were used for the fabrication of field effect transistors. The patterned electrodes defined transistors with channel length of 20 µm and channel width of 1 mm. The deposition of the active materials was accomplished by vertically immersing the substrates in the s-SWNT solution for 1 hour, afterward the substrate was rinsed by a short dipping in toluene. The substrates were then annealed for 1 hour at 120 °C to remove residual solvent. The electrical measurements were performed using a probe station connected to a semiconductor parameter analyzer (Agilent E5262A). All fabrication and characterization procedures were done in a nitrogen-filled glovebox.

**AFM microscopy.** The morphology of the deposited networks of SWNT within the channel of the FETs were imaged by Atomic Force Microscopy. AFM images were taken with a Bruker microscope (MultiMode 8 with ScanAsyst) in tapping mode with TESP-V2 probes having elastic constant k = 42 N/m, resonance frequency in the range 320–410 kHz, and tip radius less than 10 nm. The images were taken with a scan rate of 1 Hz and the resolution of 1024 lines/sample.
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6.6 References


