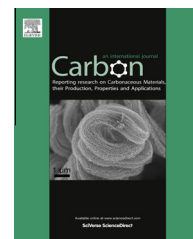


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Triton assisted fabrication of uniform semiconducting single-walled carbon nanotube networks for highly sensitive gas sensors

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ABSTRACT

Surfactants are vital for the dispersion and separation of single-walled carbon nanotubes (SWCNTs) when using chromatographic methods. However, excessive surfactant impurities left on the sorted tube surface and in solution may dramatically affect the fabrication and performance of various SWCNT-based thin-film devices. We demonstrated that compared to the widely used anionic surfactants, including sodium dodecyl sulfate, sodium dodecyl benzene sulfonate and sodium deoxycholate, the nonionic Triton surfactant was more effective not only for the enrichment of semiconducting (s-) SWCNTs, but also for preventing the tubes from severe aggregation upon network formation. Simply by spreading and rinsing treatment, a clean and homogenous network of s-SWCNTs was favorably formed between electrode channels, in which intertube contacts or junctions were well built up. The resultant sensor device showed an obviously improved electrical response to H₂S with the detection level down to 50 ppb even at room temperature, compared to those fabricated from the pristine SWCNTs and s-SWCNTs eluted with anionic surfactants. Our work provides an easy route for the sorting and application of s-SWCNTs at a low cost.

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1. Introduction

Semiconducting single-walled carbon nanotubes (s-SWCNTs) with high carrier mobilities [1,2] are considered to be ideal building blocks for single-tube based nanoelectronics and thin-film devices [3], such as chemical and biological sensors [4,5], high-performance field-effect transistors (FETs) [6,7], photovoltaic devices [8,9] and so on. Unfortunately, the directly grown s-SWCNTs always exist with metallic (m-) tubes [10]; it is still a large challenge in the direct growth of pure s-SWCNTs on a large scale for practical applications [11]. Therefore the post-synthetic approaches, such as electrical break down [6], dielectrophoresis [12], selective functionalization [13,14], density gradient ultracentrifugation [15], DNA

wrapping chromatography [16,17] and gel column chromatography, have been well developed to separate SWCNTs according to their electronic type, diameter, and chirality.

The recent progress in the sorting of s-SWCNTs by the post-synthetic methods [11,18] has inspired the efforts in developing high-performance s-SWCNT-based thin-film devices [19–21]. The electronic properties of s-SWCNTs were highly sensitive to environmental changes induced by the effects of chemical doping or charge transfer between adsorbed substances and nanotubes [22]. The devices based on the sorted s-SWCNTs have achieved the highly sensitive detection of a variety of molecules, including proteins [5], trinitrotoluene [23] and some gases [4,19] (NO₂, NH₃, H₂, etc.) due to effectively avoiding the percolation of the conductive

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pathways of m-SWCNTs [24–26], promising their applications in high-performance sensor devices. Furthermore, controlling the nanotube networks with suitable intertube connections [23,27,28] can improve the sensing sensitivity to different degrees, indicating that the response sensitivities of devices greatly depend on the inherent properties of nanotubes and tube/bundle junctions in SWCNT networks [29].

In our previous work, we achieved a m/s and few-chirality separation of SWCNTs by the procedural surfactant elution using dextran-based gel column chromatography [30]. Herein, we demonstrated that the surfactant selection was not only important for the high-efficiency enrichment of the s-SWCNTs but also for the fabrication of the nanotube network on the substrates. In contrast to anionic surfactants, such as widely used sodium dodecyl sulfate (SDS), sodium dodecyl benzene sulfonate (SDBS), and sodium deoxycholate (DOC), Triton, a nonionic surfactant, showed obvious advantages in the sorting of s-SWCNTs as well as the network formation with more isolated tubes and thinner bundles. We proposed that Triton surfactant with the large hydrophilic poly (ethylene oxide) chain played an important role in preventing the nanotubes from aggregating. Triton residues deposited between electrodes could be removed by water rinsing easily, ensuring the good intertube contacts in the network. The sensors based on Triton-eluted s-SWCNT networks therefore exhibited a high sensitivity towards H_2S with the detection level down to 50 ppb at room temperature.

2. Experimental

2.1. Enrichment and characterization of s-SWCNTs

SWCNTs prepared using the high-pressure catalytic method (HiPCO, Lot No. P0276) were purchased from Carbon Nanotechnologies Inc. 1% SDS aqueous solution was used for the SWCNT dispersion. Dextran-based gel (sephacryl S100 HR) purchased from General Electric Healthcare as the stationary phase was packed in the column. The purification, dispersion and separation of SWCNTs were performed as described in our recent report. The separation of m- and s-SWCNTs was performed by a two-step surfactant elution on a home-made gel column chromatographic machine on a large scale. 1% SDS solution was used for the enrichment of m-SWCNTs, while SDBS, DOC, Triton, and SDS solutions were used for the enrichment of s-SWCNTs. The UV-vis-NIR absorption spectra of the unsorted SWCNT dispersion and sorted s-SWCNTs were recorded on a Perkin Elmer Lambda 950 instrument. Photoluminescence spectra were obtained on the NS1 NanoSpectralyzer. Raman spectra were obtained on a Renishaw micro-Raman spectrometer. The zeta (ζ)-potentials of the s-SWCNT solutions eluted with 4% SDS, 1% SDBS, 1% DOC and 1% Triton solutions were measured with a Malvern Zetasizer NanoZS system with irradiation from a 632.8 nm He-Ne laser.

2.2. Fabrication and characterization of SWCNT networks

The networks fabricated from four surfactant-eluted s-SWCNT solutions were prepared on the substrates by the

drop-depositing method. In order to present the same volume of s-SWCNTs, the four surfactant-eluted s-SWCNT solutions were diluted with water to obtain the same tube concentration through keeping the same value (0.037) for the absorption peak at 647 nm in their absorption plots. The 0.05 μ l diluted SWCNT solution was drop-deposited on the substrates such as SiO_2/Si , glass and polyethylene terephthalate (PET). The dropped SWCNT solutions were dried in oven at 50 °C for 10 min. Next, the thin films were rinsed sufficiently with deionized water of 100 ml for three times and then were immersed in hot deionized water of 100 ml for 30 min, ensuring that most of residual surfactants could be dissolved in hot water. Finally the networks were taken out and completely dried in oven. The field-emission transmission electron microscope (Quanta 400 FEG, SEM) and atomic force microscopy (Dimension 3100, AFM) were used to characterize the morphology of the s-SWCNT networks. Current-voltage (*I*-*V*) curves were measured at room temperature with Keithley's SourceMeter 2602 system and Everbeing's PE-4 probe station.

2.3. Fabrication of SWCNT network based sensor devices

Sensor arrays were fabricated using standard lithographic techniques. First, a 500 nm SiO_2 layer was thermally deposited on a highly doped p-type (100) oriented Si wafer. Interdigitated microelectrodes (Cr/Au, 30/200 nm) were sputtered on top of SiO_2 , with a 10 μ m electrode gap. The electrode area was defined using photolithography with positive photoresist, followed by lift-off techniques. The pristine SWCNT and sorted s-SWCNT networks were fabricated by the drop-depositing method, as described in the above text. Finally, the sensors were annealed at 300 °C in a 5% H_2 and 95% Ar atmosphere, to create better contacts at the tube-tube junctions, as well as between the Au electrodes and the tubes.

2.4. Testing of s-SWCNT gas sensors

For gas sensor measurements, the chips with multiple sensor devices were Au wire-bonded on a 24-pin Cerdip, and were tested using a custom electronic test apparatus. A quartz gas flow chamber with gas inlet and outlet ports was used for gas flow through the capped and sealed sensor chip. The total gas flow rate was kept constant at 200 std. cm^3/min and regulated by mass flow controllers (MFCs, Sevenstar CS200). N_2 (purity: 99.999%) was used as both carrier and diluting gas. Analyte gases with desired concentrations were generated by diluting H_2S (purity: 99.999%) with N_2 in gas sampling bags regulated by MFCs. A custom lab-view computer program was developed to continuously control and monitor the voltage of the circuit using Fieldpoint analog input and output modules (National Instruments, Austin, TX). In the sensing test of H_2S gas, the sensors were first exposed to N_2 gas to obtain the stable baseline resistance, and, and then were switched to H_2S with desired concentration, and finally back to N_2 purge followed by short periods about 10 min to return the devices to their baseline resistances. All experiments were carried out at room temperature. The changes in the resistance of the SWCNT-based gas sensor with changing H_2S gas concentrations were recorded using a customized Labview (National Instruments) program.

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