One-step and low-temperature synthesis of the carbon nanotube with no post treatment and high purity

Xiang Li. Guo, Long Zhang, You Song. Liu and Jun Wang

1.1 Synthesis Process of SiNWs

Inourexperiment, (100)-oriented p-type silicon wafers were purchasedand cut into $2 \times 2 \text{ cm}^2$ small pieces by glass sword. A metal catalytic etching method was used to preparemonocrystalline silicon nanowire arrays. In a typical process, the piecesof the selected silicon wafers were washed by sonication inacetone and deionized water. Then the silicon wafers weredipped into HF/H₂O solution (1:10) to remove the thin oxidation layer and then dried by N₂ blow. Subsequently, the silicon waferswere immersed in a solution of 0.14M HF and 0.01M AgNO₃ for 30 s. After a uniform layer of Ag nanoparticles were coated, the wafers were immersed in the etchant solution composed of HF, H₂O₂ and H₂O (the volume ratiobeing 20:10:70) at room temperature in a sealed teflon vessel. The Si wafers were immersed in a solution of concentrated nitric acid solution toremove the excess Ag nanoparticles and then rinsed with deionized water anddriedin vacuum at 60 °C.

1.2 Synthesis Carbon Nanotubes

Carbon nanotubes were deposited in the reactor, showed in Fig 1. Undiluted hexafluoropropylene oxide (HFPO; CF₃CFOCF₂) was used as precursor gas, the content was 99.5%. The decomposed temperature of HFPO was 185 °C. The flow of HFPO is 50 sccm. The morphology of carbon nanotubes and silicon nanowires were investigated through The morphologies and structures of all the products were

analyzed by FE-SEM (Ultra 55), TEM (Libra 200FE operated at 200 kV). The Raman spectrum was taken using a micro-Raman/Photo-luminescence system (In Via).

2 Results



Figure 5S. Raman spectrum of CNTs sample

Raman spectroscopy was used to characterize the microstructure of CNTs as shown in Fig 5S. The peak at around 1,615 cm⁻¹ (G-band) corresponding to an E2g mode of hexagonal graphite is related to the vibration of sp²⁻ hybridized carbon atoms in a graphite layer. The D-band at about 1,370 cm⁻¹ can be ascribed to the vibration of carbon atoms with dangling bonds in the plane terminations of disordered graphite or glassy carbons.



Figure S2. TGA curve of CNTs prepared by CF_2 radicals and porous silicon nanowires arrays.

We further investigated the carbon content of products by the thermogravimetric analysis (TGA) shown in Figure S1. The TGA analysis shows that a major weight loss of the sample took place between 460-540 °C during the heating process in air. The lost value is about 90 wt % of the product, which indicates that the carbon content in the as-prepared product is 90 wt %. The result imply that our method can realize the synthesis of CNT with high purity and no post treatment is needed.



Figure S3. FE-SEM and TEM images of silicon (a) and (b) TEM and HRTEM images of silicon nanoparticles. (c) FE-SEM image of a cross section region of silicon

nanowire arrays. (d) TEM image of silicon nanowire scrapped from silicon substrate.



Figure S4. TEM image of nanotubes grown on porous silicon particles, the growing point shown in blue circles. The TEM image show that the silicon particles in porous silicon nanowire offer growing points for carbon nanotubes.



Figure S5. (a) FE-SEM image of porous silicon nanowires arrays after completely

reacting with CF_2 radicals and removing carbon nanotubes via ultrasonic separation; (b) TEM image of the nanotubes and carbon nanofilm; (c) TEM image of carbon nanofilm synthesized via reaction between CF_2 radicals and porous silicon. (d) HRTEM of (c) sample.