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

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1.1 Synthesis Process of SiNWs

In our experiment, (100)-oriented p-type silicon wafers were purchased and cut into $2 \times 2$ cm$^2$ small pieces by glass sword. A metal catalytic etching method was used to prepare monocrystalline silicon nanowire arrays. In a typical process, the pieces of the selected silicon wafers were washed by sonication in acetone and deionized water. Then the silicon wafers were dipped into HF/H$_2$O solution (1:10) to remove the thin oxidation layer and then dried by N$_2$ blow. Subsequently, the silicon wafers were immersed in a solution of 0.14M HF and 0.01M AgNO$_3$ for 30 s. After a uniform layer of Ag nanoparticles were coated, the wafers were immersed in the etchant solution composed of HF, H$_2$O$_2$ and H$_2$O (the volume ratio being 20:10:70) at room temperature in a sealed teflon vessel. The Si wafers were immersed in a solution of concentrated nitric acid solution to remove the excess Ag nanoparticles and then rinsed with deionized water and dried in vacuum at 60 °C.

1.2 Synthesis Carbon Nanotubes

Carbon nanotubes were deposited in the reactor, showed in Fig 1. Undiluted hexafluoropropylene oxide (HFPO; CF$_3$CFOCF$_2$) 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 (InVia).

2 Results

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 E\(_{2g}\) mode of hexagonal graphite is related to the vibration of sp\(^2\) 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.
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 S2. TGA curve of CNTs prepared by CF₂ radicals and porous silicon nanowires arrays.

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 scraped from silicon substrate.

Figure S4. TEM image of nanotubes grown on porous silicon particles, the growing point shown in blue circles. The TEM image shows 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.