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Synthesis and characterization of ultra-long straight carbon wires

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ABSTRACT

Ultra-long straight carbon wires with few microns in diameter and centimeters in length were synthesized in large scale through a controllable one-step chemical vapor deposition (CVD) method. The ratio of axial length to diameter more than 1000:1. The scanning electron microscope (SEM) and high-resolution transmission electron microscopy (HRTEM) images prove the layered structure surrounding the wire center, and the energy dispersive spectra (EDS) confirm the main component of the as-obtained carbon wires is carbon. Room temperature Raman spectrum presents two distinct Raman peaks of 1351 cm^{-1} (D band) and 1586 cm^{-1}(G band), which is consistent with carbon crystalline structures. I-V curves exhibit typical resistance characteristic, which can be used as electronic polar or mechanic probe to operate nanostructures.

1. Introduction

The unique features of one dimensional carbon nanostructures including ultrahigh strength [1,2], high electrical conductivity [3] make it widely used in nanolithography [4], probes [5,6], field emission device [7] and transistors [8]. However, most situations, the hollow nanotubes were easily synthesized rather than solid nanowire. The synthesis of large-scale solid carbon wires is still a challenge for mass production [9]. The solid carbon micro wires with large ratio of length to diameter will be used as mechanic probe to operate small size structures due to high hardness and can be used as electronic polar to construct micro circuit. Catalysts are a very important issue for the preparation of carbon nanostructures [10]. In this paper, we tried to use Si as the catalyst. The ultra-long, straight, parallel-growth carbon wires with a ratio of axial length to its diameter more than 1000:1 were synthesized. The carbon wires were obtained through heating the mixed precursors of Si, SiO_2 and graphite powders with a controllable one-step CVD method. The I-V curves of solid carbon microstructure exhibit typical low-resistance characteristic. It proved that can be used as electrical polar to construct or measure the nanowire-based electricity.

2. Experimental

The carbon wires were synthesized by a CVD method in a conventional horizontal furnace with a quartz tube using porcelain boat as growth container. Si, SiO_2 and graphite used as precursor and mix with the molar ratio of 1:1:10 in the center of furnace. Prior to a rapid heating to 1400 °C within 20 min and being kept at this temperature for 3 h with the Ar/H_2 (10%) flow of maintaining at 10 sccm, the quartz tube was purged with high-purity Ar (90%)/H_2 (10%) at a constant flow rate of 50 sccm for 1 h to remove air. After the furnace cool down to room temperature, we have the ultra-long carbon wires on the surface of powder kept in the porcelain boat. Growth direction of wires is parallel to the flow direction of source gas.

The morphology was characterized by TEM (Tecnai G2 F20), SEM (Zeiss SUPRA 55) equipped with an EDS. Optical images were captured by an optical microscope (Olympus BX51M). I-V curve was measured by a microprobe operation system with Keithley (4200). Raman spectrum was obtained by using a DPSS laser (CVI Melles Griot 58 GLS) as a pumping source. The lasers beam was focused on the sample by a confocal microscopy system with a 100X objective lens and the Raman results were collected and analyzed using μ-Raman systems.

3. Results and discussion

Fig. 1(a) shows dark-field image of the carbon wire under white light illumination. We also found that the carbon wire adheres to a small ball with metallic shininess. When Si powder is removed from the precursor, no carbon wires could be synthesized. Si acts as the catalyst for the growth. Further EDS result proves the major component of this ball is silicon. Fig. 1(b) shows the optical image of an ultra-long and straight carbon wire, which is captured by a

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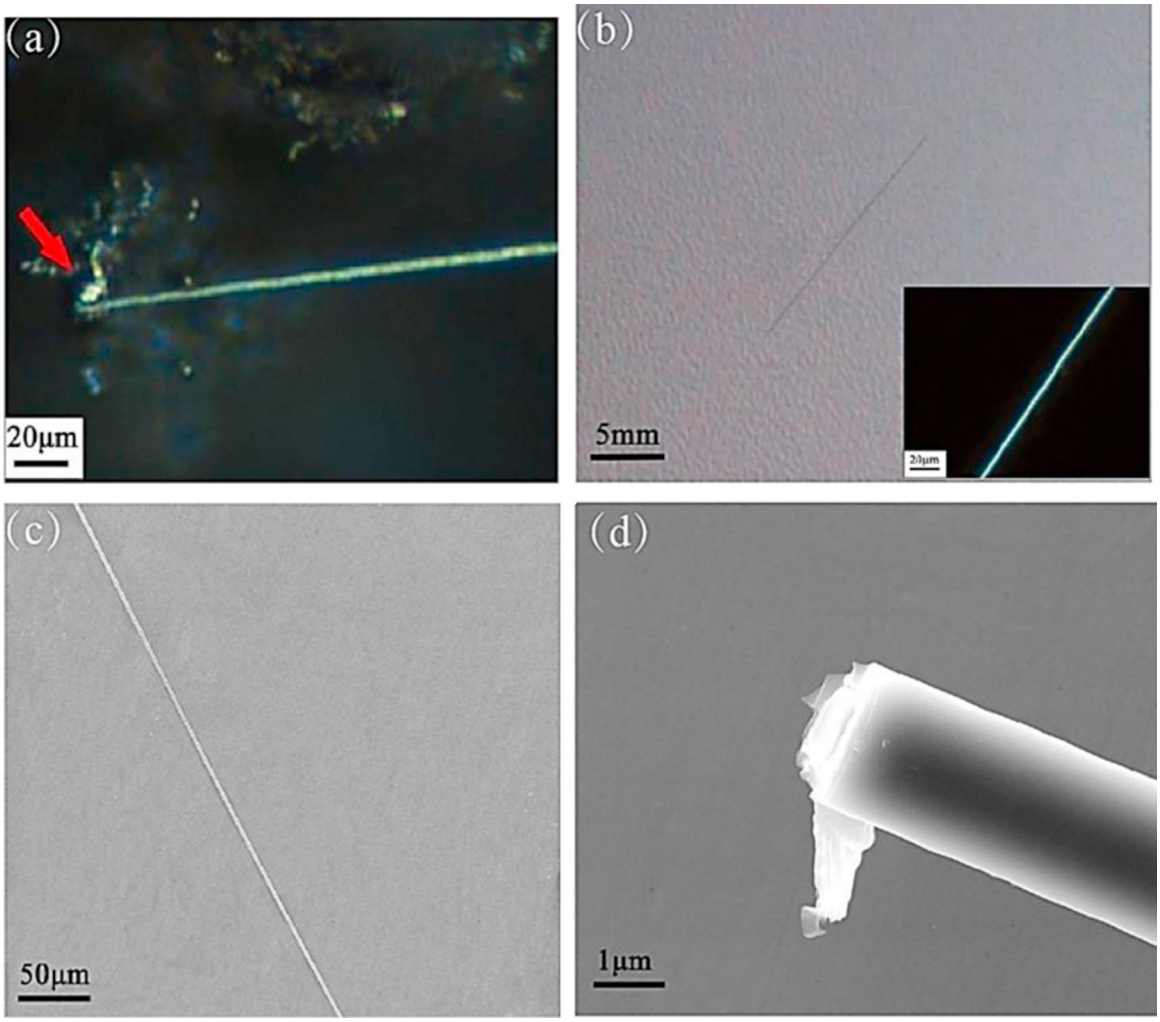

Fig. 1. (a) Dark field image of the carbon wire on powder by optical microscopy. The red arrow is a silicon ball. (b) The optical and dark field images (inset image) of single carbon wire measured under white light illumination. (c) Low-magnification SEM image of a single typical carbon wire. (d) High-magnification SEM image of the cross-section. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

CCD. The length of the carbon wire is more than 1.5 cm. The diameter of the wire is measured every 100 μm and the average diameter is 2.6 μm. The maximum diameter is 2.8 μm and the minimum is 2.5 μm. The inset shows the dark-field image of a single carbon wire with about 2.6 μm in diameter and exhibits high uniformity. SEM image exhibits the uniform growth of carbon wire, as shown in Fig. 1(c). The corresponding SEM image of the cross-section in Fig. 1(d), which proves it is a kind of multilayer structure surrounding the wire center.

Fig. 2(a) shows the high resolution image of carbon wires. Fig. 2 (b) shows the EDS table of different selected portion of carbon wire as red mark in Fig. 2(a). From the EDS data, we believe that the silicon is used as catalyst for the growth of carbon wire, which is further confirm by XRD pattern as shown in the inset of Fig. 2(a). XRD pattern exhibits that all peaks of the XRD pattern are in agreement with the standard values of the hexagonal phase graphite (JCPDS Card no. 41-1487). At first core of silicon is formed near fusing temperature, which is used as catalyst for carbon wires growth. Therefore, part II is the nearest part to the core layer with the highest atomic ratio of element Si, which is greater by 14% than part I and part III. HRTEM image indicates the distribution of carbon is parallel bending towards the interior and the inner silicon core is wrapped with multilayer of the carbon. The interplanar spacing is 0.334 nm, which is consistent with the (002) crystal orientation. Fig. 2(d) shows the STEM image of the carbon wires and a thin deciduous layer of carbon can be found on the end surface (red rectangle). The inserted selected area electron diffraction (SAED) pattern in Fig. 2(d) suggests that the thin deciduous layer is a typical hexagonal lattice structure of carbon, which confirms the XRD result presented above. These consequences prove that Si acts as the catalyst in the growth of these ultra-long straight carbon wires.

The typical Raman spectrum of the as-synthesized carbon wires is composed of two peaks centered at around 1351 cm^{-1} (D band) and 1586 cm^{-1}(G band) as shown in Fig. 3. Small crystallite size is expected due to the relative high intensity of D-band [11]. Large integrated intensity ratio I_D/I_G for the D band and G band characterize high defect quantity in carbon wires [12], and shielding effect from carriers of defect lead to the decrease of the frequency of G* band at 2900 cm^{-1}. However, a small peak is also observed at 523 cm^{-1}, which is agreement with silicon. In view of the sample is disperse on glass slide, so silicon peak comes from the wire.

Electrical properties of these carbon wires are examined to investigate its potential application in micro electrical circuits. Fig. 4(a) shows the I-V curve of a single CdS nanowire using carbon wires as electrodes. I-V curve is measured under periodic background light illumination of a microscope. The response to light is sensitive to a very low contact resistance between CdS nanowire and carbon wire . The inset of Fig. 4(a) presents a typical resistance