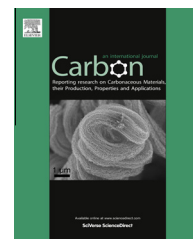


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Controllable synthesis of single- and double-walled carbon nanotubes from petroleum coke and their application to solar cells

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ABSTRACT

Single- and double-walled carbon nanotubes (SWCNTs and DWCNTs) have been controllably synthesized by an arc discharge in different atmosphere using petroleum coke as carbon source. The morphology and properties of two kinds of carbon nanotubes (CNTs) synthesized with Fe as catalyst were characterized by scanning electron microscopy, high-resolution transmission electron microscopy, Raman spectroscopy, energy dispersive X-ray spectroscopy, X-ray photoelectron spectroscopy, UV–visible spectroscopy, inductively coupled plasma optical emission spectrometer, thermogravimetric analysis and infrared spectroscopy. In the He gas atmosphere only SWCNTs were found to be synthesized by arc discharge in contrast to the case in Ar gas atmosphere in which only DWCNTs were formed. In addition, properties of solar cells based on both kinds of CNTs and n-type Si are examined under illumination of light emission diode (LED). It is found that the performance of solar cells depends significantly on the type of CNTs, i.e., SWCNTs-based solar cells show better performance under LED illumination with wavelengths in the range of 400–940 nm than the case of DWCNTs which exhibit high performance under illumination of the 1310 nm infrared light.

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1. Introduction

The petroleum coke as a by-product from refining of petroleum residuals consists of condensed aromatic clusters [1], being used in a furnace or boiler for the generation of heat or “lighting up” facility in many coal-fired power plants. It also contains high content of contaminants such as metal compounds and particularly sulfur which forms sulfur dioxide upon combustion, which make petroleum coke very cheap. On the other hand, the byproducts of petroleum industry, such as pitch and coke, have great potentials as

feedstock in preparing various kinds of carbon materials [2,3]. Carbon nanotubes (CNTs) were first discovered by Iijima in 1991 [4], and research in this field has grown exponentially due to their unique mechanical, thermal, electrical and optical properties [5,6]. Consequently, application of CNTs in electronics, high strength composites, chemical, biosensors, catalyst support, fuel cells, solar cells and many other fields have been pursued vigorously [7–12]. Specially, there is a recent growing interest in developing CNTs–silicon heterojunction solar cells which are typically made by depositing a transparent CNT film on the surface of an n-type Si

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wafer to form CNT–Si junction [13–16]. Compared with traditional Si solar cells, the CNT–Si heterojunctions solar cell can be used to convert infrared light into electricity [17].

To date, the predominant methods used for the synthesis of CNTs are arc discharge and chemical vapor deposition (CVD). The arc discharge as a traditional method is usually used to synthesize the CNTs with high crystal structure, although the CVD method can also be used to synthesize high-quality CNTs recently [18]. The arc discharge method involves using two high-purity graphite rods as electrodes that are placed in a noble gas atmosphere. In addition to graphite, there has been promising research into the use of other cheap materials such as coal, pitch, oil residue as source materials [19–21]. In our previous work, we have demonstrated the feasibility of preparing CNTs from pitch [22]. Compared with the case of pitch, the content of aromatic hydrocarbons in the petroleum coke is much higher, which may be beneficial for synthesis of CNTs. In this work, our results indicate that SWCNTs and double-walled carbon nanotubes (DWCNTs) can be controllably synthesized from petroleum coke by an arc discharge method. Moreover, application of obtained CNTs for solar cells has been further studied. The configuration of solar cells consists of Si and the obtained SWCNT and DWCNT films, and their performances are investigated under different wavelengths of light emission diode (LED) illumination. It is found that the performance of solar cells depends on the type of CNTs synthesized from petroleum coke. SWCNTs-based solar cells show much better performance under short wavelength (400–940 nm) illumination than the case of DWCNTs which exhibit high performance under infrared illumination (1310 nm).

2. Experimental

2.1. Synthesis of SWCNTs and DWCNTs from petroleum coke by arc discharge

In this study, the arc discharge method was adopted to produce SWCNTs and DWCNTs from petroleum coke which was by-product of delayed coking processing from Lanzhou in China. To make an coke-derived electrode, the coke sample was crushed and sieved to 100–200 mesh, and further mixed with Fe powder (100 mesh) as catalyst in the weight ratio of 1:2. Then the mixture was filled into hollow cylinder carbon rods with bottom at one side (5 mm inside diameter, 8 mm outside diameter, 70 mm in length and 50 mm in depth). The CNTs were prepared by electrically arcing carbon rods in high-purity gas atmosphere including N₂, He and Ar in a stainless steel arc discharge chamber with an inner diameter of 165 mm and a height of 410 mm. The anode was a coke-derived carbon rod and the cathode was a high-purity graphite electrode (20 mm in diameter, 38 mm in length). In order to facilitate the collection of CNTs and improve the purity of the CNT samples, a wire net was placed on the top of the two electrodes in the chamber, and the distance between the wire net and the electrodes was about 5 cm, which was similar to the case mentioned previously [19]. The buffer gas pressure was controlled in the range of 0.04–0.05 MPa during

the arc discharge process. The DC voltage and current for arcing were controlled at 16–18 V and 70–80 A, respectively. The distance between the two electrodes was maintained at about 1–3 mm by manually advancing the anode that was consumed during the experiments. The weight of the graphite cathode remained the same before and after the arcing experiments. After the reaction was finished, the film-like samples were collected from the wire net and examined by using scanning electron microscopy (SEM, FEIQuanta200F), high-resolution transmission electron microscopy (HRTEM, FEIF20), energy dispersive X-ray spectroscopy (EDX) coupled with HRTEM, Raman spectroscopy (514.6 nm laser, Horiba Jobin Yvon T64000), X-ray photoelectron spectroscopy (XPS, Thermo Fisher K-Alpha), infrared spectroscopy (MAGNA-IR 560 E.S.P), UV spectroscopy (Yoke, UV759/723), inductively coupled plasma optical emission spectrometer (ICP-OES, OPTIMA7000) and thermogravimetric analysis (TGA, NETZSCH STA409PC).

2.2. The fabrication of CNTs-solar cell

Two kinds of CNTs including SWCNTs and DWCNTs were obtained from coke through above mentioned process. The solar cells were fabricated by directly picking up SWCNT or DWCNT films onto the single crystal n-type silicon with resistance of 5–10 $\Omega \text{ cm}^{-1}$ with a window ($0.2 \times 0.2 \text{ cm}^2$) on which an silver paste electrode with thickness of about 200 μm was separated by a 100 nm SiO₂ insulator layer. The schematic illustration of solar cell devices is shown in (Fig. S1). Current–voltage (*I*–*V*) characteristics of solar cells were detected by using a semiconductor parameter analyzer (Agilent B2902A) under illumination of LED light sources with different wavelengths (400, 520, 600, 630, 730, 850, 880, 940 and 1310 nm).

3. Results and discussion

Fig. 1a and b show the macroscopic images of film-like deposits synthesized from coke during arc discharge carried out in He and Ar atmosphere, respectively. According to the images, it is found that the film-like samples are very thin and nearly transparent. The yield of film-like sample is affected by the gas atmosphere during arc discharge, and the samples synthesized from petroleum coke in the He and Ar atmosphere conditions are much higher than the case prepared in the N₂ atmosphere. SEM has been further used to study the as-formed film-like deposits. Fig. 1c and d show two typical SEM images of film-like samples obtained from arcing coke with Fe as catalyst under He and Ar atmosphere, respectively, revealing that all the coke-derived film-like deposits were cotton-like threads or bundles, consisting of high-density CNTs. The coke-derived CNTs were further examined by HRTEM, as seen in Fig. 2. The morphology of CNTs prepared in He condition is verified to be SWCNTs according to the HRTEM observation in Fig. 2a, and over 95% CNTs are SWCNTs. By comparison, Fig. 2b indicates clearly that the CNTs synthesized in the Ar atmosphere are almost DWCNTs with purity of 97%. It is necessary to note that the purity evaluation of SWCNTs and DWCNTs are based on a lot of

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