

Quantitative limitation of active site and characteristics of chemical oxidized well-aligned carbon nanotubes

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Available online 14 July 2007

Abstract

For many years, acid treatment processes have been used to modify substances for their applications on energy storage devices, such as electrochemical capacitors, fuel cells, etc. It is obvious that the increase of surface area and functional groups has beneficial influence on the amount of reactant adsorption. However, the quantitative limitation of active site has been poorly discussed until today. In this experiment, nitric acid (2 M and 14 M) was introduced at 90 °C over different periods of time to oxidate the well-aligned multi-wall carbon nanotubes (MWNTs), which were synthesized directly using carbon cloth (CC) as the substance. Quinoidal functional groups on active CNTs, such as -COOH and -OH, were found to be the result of chemical reaction. Pt catalyst, which was extracted from $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$, was deposited onto the carbon nanotubes using electroless plating in chloroplatinic acid solution. The extent of Pt adsorption was measured and was substantially larger than the as-prepared CNTs. It was found that the state of quantitative limitation existed in both 2 M and 14 M systems. A model was also developed to illustrate the limitation in the active site due to the chemical oxidation.

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Keywords: Modify; Functional group; Adsorption; Nanotube; Limitation

1. Introduction

Since carbon nanotubes were first discovered by Iijima [1], it has attracted much attention owing to its novel properties [2–4]. Carbon nanotubes (CNTs) are known to possess exceptional mechanical, electrical and thermal properties, because of their high strength, low density, and high electrical and thermal conductivity [5]. There are numerous potential applications for CNTs, such as flat panel display [6], sensors [7], nanoscale devices [8], vehicles for hydrogen storages [9] and electrochemical energy-storage devices [10]. Especially regarding its application on energy-storage devices, it has drawn much attention to the change in the surface characteristics. It is a common practice to convert the surface bonding into some kinds of functional groups using chemical treatment with

$\text{H}_2\text{SO}_{4(l)}$, $\text{HNO}_{3(l)}$, $\text{KOH}_{(l)}$, etc. [11–13]. Recently, electrochemical process demonstrated that the functional groups are beneficial towards the uniform separation of deposition [14] and the extent of ion-adsorption [15]. Unfortunately, up to now, the limitation of active site related to chemical oxidation has not been discussed quantitatively.

At the moment, we are aiming at directly fabricating carbon nanotubes onto carbon cloth as the electrodes, and quantitatively illustrate the limitation of chemically oxidized multi-wall carbon nanotubes in the active site, which is related to the adsorption of ions to charge. Physical characteristics of the electrodes will be measured and demonstrated.

2. Experimental

Detailed description of the synthesis of multi-wall carbon nanotubes (MWNTs) by microwave plasma-enhanced chemical vapor deposition system (MPECVD), without substrate bias, was reported previously [16]. In this work, carbon cloth (CC,

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typically 2 cm × 2 cm in size) was used as a substrate on account of the flexibility and stability on higher operating voltage. In order to synthesize carbon nanotubes onto CC successfully, we firstly coated Fe as the catalyst on CC by magnetic sputtering. Then the synthesis of carbon nanotubes was processed. For the growth, a mixture of hydrogen and methane, with the gas flow of 90 sccm and 30 sccm respectively, was introduced into the chamber at the pressure of 10 Torr and then it was operated at 300 W. For electrochemically oxidized carbon nanotubes, MWNTs electrode was treated with 2 M and 14 M HNO₃ at 90 °C for different periods of time (0–48 h). Then it was flushed with deionized water repeatedly and purged with N₂ for 10 min.

For the deposition of Pt, the prepared electrodes were immersed in a mixture, which comprise H₂PtCl₆ · 6H₂O, PVP-4000 and ethylene glycol, diluted with acetone. PVP is a protection substituted to limit the particle growth spacing. H₂PtCl₆ · 6H₂O was used as the precursor of Pt deposition. It was operated at 160 °C for 3 h, followed by filtration with acetone and sintered at 250 °C for 1 h. Measurements were carried out as follows.

The microstructure was respectively observed by means of the JEOL JSM 6500 and JEOL JEM 4000. XPS (ESCA PHI 1600 using an Mg K α X-ray source) and EDX (JEOL

JSM6500F) measurements were carried out for the identification of Pt particles. The functional groups on the surface of oxidized MWNTs can be determined by FTIR on PROTEGE 460 series.

3. Results and discussion

Fig. 1(a) shows SEM image of the as-prepared MWNTs fabricated densely onto the carbon cloth. It is well-aligned; having a length and diameter of approximately 20 μ m and 20 nm, respectively. The TEM image shows hollow structure with amorphous carbon outer surface (outside) and crystalline layers in the inner surface, as can be seen in Fig. 1(c).

Unfortunately, even though the surface area of CNTs is high, it still cannot be completely utilized by physical deposition since this method usually makes the deposition uneven and concentrated on just a part of the surface. On the contrary, the deposition could become uniform by means of the chemical polyol procedure due to the good fluidity and contact with overall area. However, the hydrogen dangling bond on the top surface of the raw MWNTs makes them relatively inert and also makes the agglomeration of particles large in size. In practice, it is important to improve the adhesion efficiency, to make them

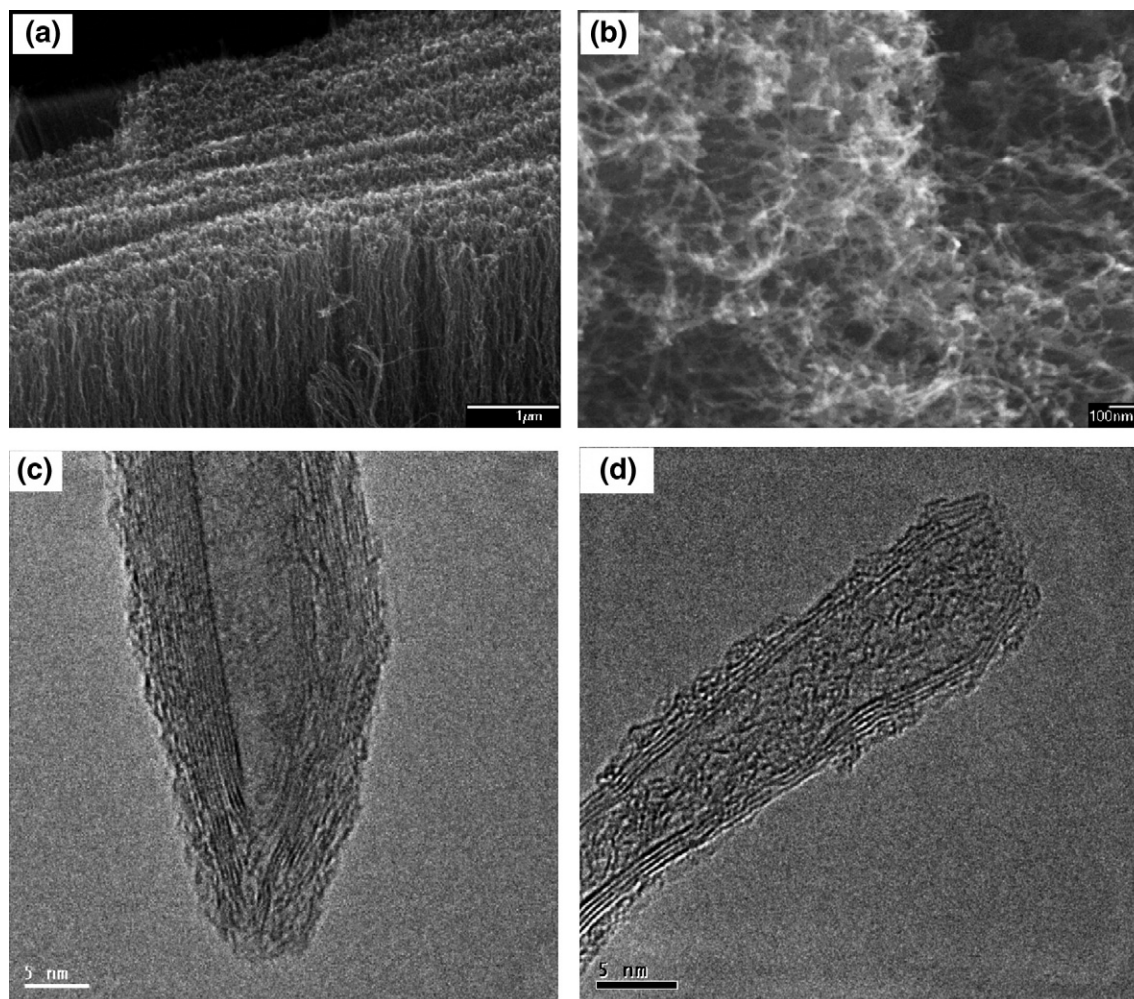


Fig. 1. Typical SEM and TEM images of the MWNTs, synthesized by MPECVD, showed (a, c) before and (b, d) after the chemical oxidation (HNO₃, 14 M).

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