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Purification of carbon nanotubes grown by thermal CVD

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Abstract

We show the results of a set of purifications on carbon nanotubes (CNT) by acid and basic treatments. CNTs were obtained by thermal decomposition of camphor at 850 °C in a CVD growth system, by means of a growth process catalyzed by iron clusters originating from the addition of ferrocene in the precursors mixture. The purification procedures involved HNO_3, H_2SO_4, HSO_3Cl and NaOH for different process temperatures.

As-grown CNTs showed a consistent presence of metal catalyst (about 6 wt%), evidenced by TGA. The purification treatments led to a certain amount of opening of the CNT tips, with a consequent loss of metal catalyst encapsulated in tips. This is also confirmed by BET analysis, which showed an increase of the surface area density of CNT after the purification.

FT-IR and XPS revealed the presence of carboxylic groups on the CNT surface chemically modified by the harsh environment of the purification process.

Among the various treatments that have been tested, the 1:3 solution of nitric and sulphuric acid was the most effective in modifying the CNT surface and inducing the formation of functional groups.

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Keywords: Carbon nanotubes; Acid/basic treatments; Functionalization

1. Introduction

Carbon nanotubes (CNTs) grown by arc discharge, laser ablation of graphite and chemical vapor deposition (CVD) contain impurities, most notably metal catalyst particles and in some cases unwanted carbonaceous byproducts (such as amorphous carbon, fullerene, etc.) [1–4]. A CNT grown by catalytic CVD usually keeps the catalyst metal particle that allowed the growth in its tip [5,6]. This could significantly affect the CNT properties. Metal particles could also be observed in CNT bodies, as a result of the catalytic growth process itself. Hence, the purification is a needed and crucial step in order to open the CNT tips and remove the metal catalyst. This challenge is still far from an exhaustive solution, for the troubling step is to get rid of the undesired byproducts without damaging the CNT structure [7–9].

The purpose of this work is to test the reaction of the CNT material grown by the catalytic thermal CVD described in Refs. [10,11], to a series of different purifications by acid and basic treatments, in order to open the CNT tips and remove the metal catalyst particles, and lead to the formation of functional groups (such as carboxylic group) which are essential in the formation of covalently bonded adducts of biological molecules with CNTs [12].

2. Experimental

Multi-walled CNTs (MWNT) were grown by thermal CVD at 850 °C in a horizontal quartz tube housed in a cylindrical furnace. An inert gas flow carried the gas mixture of carbon precursor and metal catalyst towards the center of the furnace, where gas pyrolysis led to the deposition of a CNT layer on the substrate. The carbon

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