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Low temperature electron spin resonance investigation on SWNTs after hydrogen treatment

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Abstract

We report a study of hydrogen adsorption experiment on SWNT samples deposited by chemical vapor deposition (CVD) and investigated with Electron Spin Resonance spectroscopy (ESR) before and after hydrogen treatment. The ESR investigation was performed at room and low temperature (4–30 K). We found that ESR technique is very useful to investigate paramagnetic centers and their interaction with hydrogen.

The morphology and structure of the samples were characterized by Micro-Raman spectroscopy. Subsequently the thermal stability was investigated by thermogravimetric analysis (TGA) testing. Brunauer Emmett Teller (BET) analysis was used to calculate the specific surface area and porosity of the as grown samples. All characterizations showed a small presence of residual Fe impurity (3.3 wt.%).

Keywords: Carbon nanotubes; Chemical vapor deposition; Hydrogen storage; ESR

1. Introduction

The world is becoming more and more conscious about its consumption of fossil fuels and about consequent environmental problems. As a result, interest towards possible alternative sources of energy is rapidly increasing. Hydrogen is expected to be the best candidate [1] as a renewable and clean energy carrier. The current ways of storing hydrogen are gas bombs, refrigerated (at 77 K) tanks and adsorbed in solids such as metal hydrides.

Recently, tremendous interest has been aroused by the discovery of the hydrogen adsorption capacity in carbon materials [2], such as nanoporous carbon or nanofiber and, in particular, carbon nanotubes (CNTs) [3]. CNTs show unique physical/chemical properties for many potential applications [4] and their low density and high porosity makes them a potentially useful and safe solid hydrogen storage system [5]. However the reported amounts of hydrogen uptake in CNTs varies significantly depending on the research groups, and the mechanism lying behind the gas interaction with the carbon layer is not yet fully understood [6].

In this study, we used electron spin resonance as a toll to investigate the effect of hydrogen adsorption in SWNTs grown by chemical vapor deposition (CVD) on the various properties of the ESR signal, such as spin density and relaxation time.

2. Experimental

The single wall carbon nanotubes (SWNTs) investigated in this report (average diameter <2 nm; length of a few microns) were provided by Thomas Swan and Co. They have been deposited by CVD and have a 70–90% purity level in weight. No attempt to perform further purification or catalyst extraction was made in order to prevent large-scale modification of the material [7].

As-grown samples were characterized by electron microscopy (not reported in this work) and Raman spectroscopy (MicroRaman Renishaw Ramascope, 514.5 nm excitation) in order to check the amount of SWNTs.

The thermal degradation of the samples was investigated by means of a thermogravimetric analysis (TGA) 2050 balance

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