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TEM studies of conglomeration of single-wall carbon nanotubes under atomic deuterium interaction

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Relatively little experimental activity has been devoted to the interaction of atomic hydrogen with single-walled carbon nanotubes (SWNTs). An important question is how the atomic and molecular deuterium treatment affect the structural and chemical evolution of SWNTs at different temperatures. In order to elucidate this point we have used the combination of X-ray Photoelectron Spectroscopy (XPS), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). Our forthcoming publication (Ref. 1) deals with this subject. In this work we present TEM data dealing with the question of how SWNTs are affected by prolonged interaction with atomic deuterium (D).

The material, HiPco (Carbon Nanotechnologies), was first dispersed in isopropanol in an ultrasonic bath and was subsequently deposited onto Si(100). Both heating of SWNTs and their interaction with deuterium were performed in situ in a quartz cell, part of a separate UHV glass system [2], and maintained at 273 K. The SWNT samples were exposed to D produced by the thermal dissociation of D_2 on a hot W filament, its temperature, T_f, being kept at 1020 K, and a deuterium pressure of 0.005 Torr. All gas-solid interactions were monitored in situ by Thermal Desorption Mass Spectrometry (TDMS). See Refs. 2, 3 for a description. TEM examination has been realized *ex situ* in two ways: (a) the SWNT material, scraped from the silicon substrate samples, was analysed before and after the D treatment procedure, (b) a selected SWNT sample was investigated in cross-section in order to reveal the distribution of the SWNT material as exposed to D without the scraping induced deformation of the SWNT layer.

The results show that prolonged, low atomic deuterium density gas phase treatment leads to a conglomeration of bundles of SWNTs through a “patching and tearing” mechanism [4], forming large diameter carbon ropes of square and triangular cross-section covered by nano-aggregates of graphite material. A cross-sectional TEM image (Fig.1a) discloses the existence of large carbon ropes with varying diameter, forming a carbon nanotube layer of thickness between 200 and 600 nm. TEM images of the scraped SWNT material disclose the formation of SWNT ropes 40 – 80 nm in diameter (Fig. 1c) in addition to bundles of 20 – 25 nm in diameter (Fig. 1b) , which were also present in the pristine material prior to atomic deuterium treatment. Enlargements of the selected rope areas in Fig. 1c (1 and 2) are presented in Fig. 2 together with a geometric analysis of their cross-sectional shape. For a model consideration we have taken the SWNT bundle diameter to be 25 nm as we found in Fig.1b. Using the simple analytical formulas, modeling the cross-sections of ropes 1 and 2 (Fig. 2), for the corresponding values of the square (R_s) and triangular (R_t) cross-section diagonals we arrived at 61 and 47 nm, respectively. These values are close to those as measured directly by TEM (see images 1 and 2 of Fig. 2), R_s = 60 nm and R_t = 48 nm, respectively.

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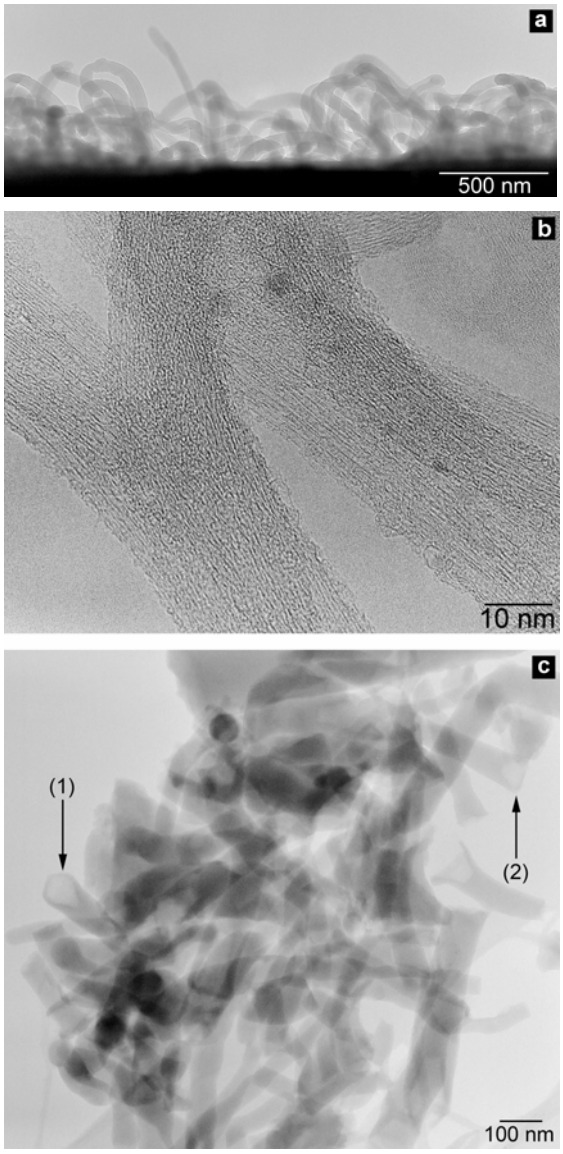

Figure 1. (a) Cross-sectional TEM image of the SWNT sample after its exposure to atomic deuterium for 60 min., (b) and (c), high- and low-magnification TEM images of the SWNT material scraped from the sample presented in (a), respectively. Low-magnification TEM image (c) shows thick ropes of bundles characterised by square- (1) and triangular-cross-sections (2).

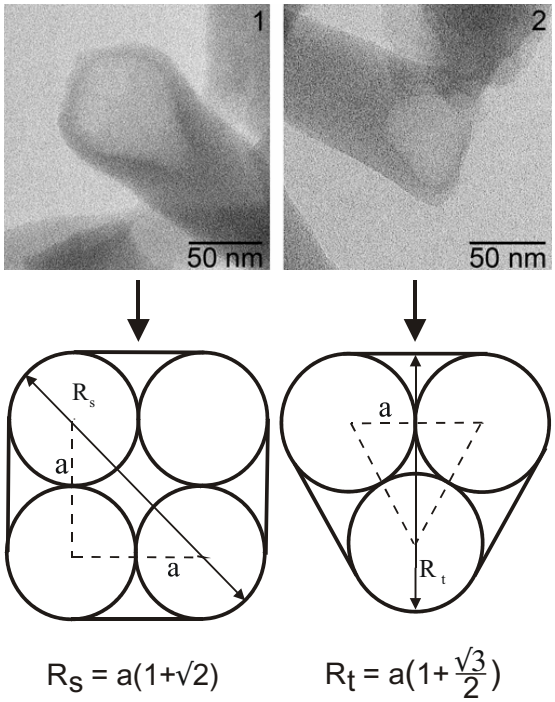

Figure 2. The analysis of the cross-sectional shapes of two SWNT ropes selected in Fig. 1c. The TEM image magnifications of the square- (1) and triangular- (2) cross-section areas are shown on top. Below are presented their schematic model approximation together with, placed in the bottom, the formulas relating square- (Rs) and triangular- (Rt) cross-section diagonals of the corresponding SWNT ropes to the SWNT bundle diameter (a).

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