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Synthesis and Characterization of Carbon Nanotubes Via Spray Pyrolysis Method

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Carbon nanotubes (CNTs) have a distinctive shape with a nanosized diameter and high aspect ratio [1]. Other features include excellent mechanical properties, high thermal conductivity, high electrical conductivity, etc. [2,3]. CNTs have been intensely applied to many fields, including composites, in recent years by exploiting their geometry and excellent properties [4,5]. Because of this enormous application potential, several methods have been reported for producing large quantities of quality CNTs at a low cost. Such methods include: arc discharge, laser evaporation and chemical vapour deposition CVD. Synthesis of CNTs by CVD methods have proved to be more controllable and cost efficient than arc discharge or laser evaporation methods. Spray pyrolysis is a modification of the CVD method and is extensively used for the large-scale production of multi-wall carbon nanotubes (MWCNTs) at the lowest cost.

The spray pyrolysis process was used to synthesis of multiwalled carbon nanotubes (MWCNTs), a quartz tube, used as a solution atomizer by injection syringe system. The overall tube dimensions had an internal diameter of 9 mm and a length of 500 mm. A cylindrical furnace (Thermo scientific) with a high precision temperature controller (±1 °C) heated the tubing. The solution feed time was kept constant for 15 min for all experiments. Fifty millilitres of toluene (J.T. Baker, 100%) and 1.00 g of ferrocene (Aldrich, 98%) were placed in a glass container. Argon (Praxair, 99.99%,) was used as the carrier gas; a mass flow controller at 17 cm^3/s regulated the flow rate. An argon/toluene/ferrocene mixture was fed into the quartz tubing after the furnace temperature was set at 800 °C.

Afterwards, the black film of MWCNTs that formed at the inner surface of the quartz tubing was mechanically removed with a brush and was analyzed by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and Raman spectroscopy.

The SEM images taken from the middle section of the quartz tube after the spray pyrolisis process, the figure 1a shows a large amount of nanotubes husk (fiber morphology) and were perpendicularly aligned with the quartz tube. The lengths of the CNTs were estimated as being greater than 1300mm and figure 1b shows SEM micrographs of randomly oriented CNT network grown at optimized conditions as mentioned before.
Based on the above results, it can be considered that spray pyrolysis method with a ratio 50ml of toluene/1ml of ferrocene at 800°C was appropriate for preparation of CNTs. For this reason, morphologies and structures of CNTs synthesized at this optimal condition were further characterized by TEM and HR-TEM. Figure 2, shows a high magnification image of an individual well graphitized CNT, revealing that its outer and inner diameters were 39 and 9 nm [6].

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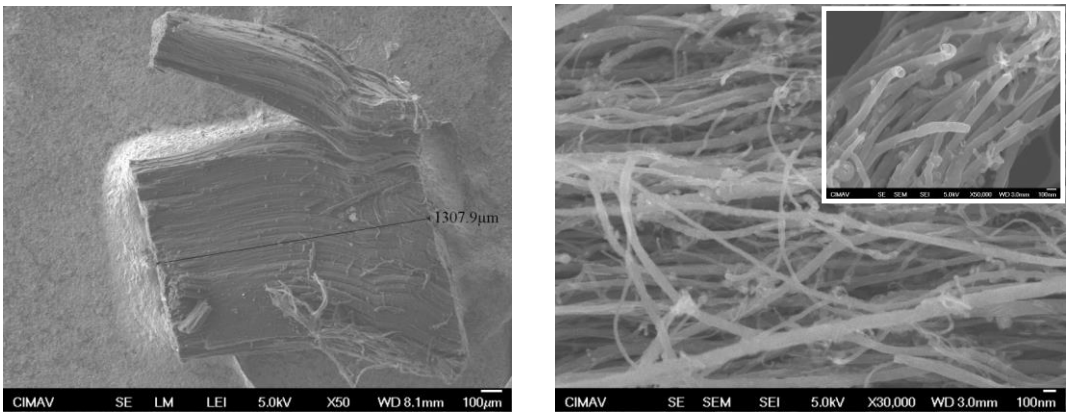

Figure 1. SEM images of nanotube films grown and the lengths of the CNTs, obtained by spray pyrolysis method

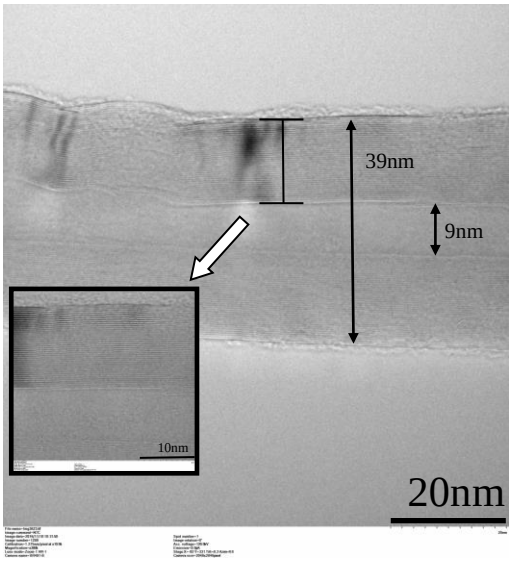

Figure 2. TEM and HRTEM images of product samples resulted from spray pyrolysis method under the optimal condition.