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SYNTHESIS OF THIN WALL MULTI-WALLED CARBON NANOTUBES BY CATALYTIC DECOMPOSITION OF HYDROCARBON USING METALLOPHTHALOCYANINE AS CATALYST

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Temperature dependence on synthesis of carbon nanotube by thermal catalytic decomposition of hydrocarbon was investigated. Benzene, thiophene, and iron(III)phthalocyanine are used for synthesis. As a result, the thickness of 20 nm, 5 to 10 layers solid structured carbon nanotube was obtained.

Keywords: carbon nanotube; chemical vapor deposition; electron microscopy; catalytic properties

INTRODUCTION

Carbon nanotube [1] is hollow body carbon fiber, which respects an application such as adsorbent of hydrogen and other molecules [2]. For practical use, pure carbon nanotube is required in the step of synthesis owing to difficulty of purification.

Yumura and Ohshima pointed out the possibility of catalytic decomposition of hydrocarbon in the continuous reactor [3]. From detailed investigation, growing process of carbon nanotube is roughly divided into two processes, fast catalytic growth of carbon nanotube core in the first, and

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slow thicken in the second. In fact, the most of synthetic examples of thin, including single wall carbon nanotube is achieved by higher flow rates [4].

In contrast, the authors planned reaction temperature lowered to reduce reaction rate of decomposition. The experiment was based on the mass production flow reactor developed by Ohshima [3], and the reaction temperature was lowered from 1200°C. As a result, 5 to 10 layer carbon nanotube was synthesized with a diameter about 20 nm. This carbon nanotube has wide bore and thin wall, which will suit for adsorbent of larger molecules.

EXPERIMENTAL

Flow reactor was shown in Figure 1. A quartz tube was placed in of tubular tantalum electric furnace. 5 ml of benzene and 1 g of thiophene (Wako GR) were mixed and placed in bubbler. At an ambient temperature of 30°C, 0.9 g of hydrocarbon was consumed. The hydrogen gas used as carrier was divided into two pipelines, one was for bubbler, and the other was for dilution. Each flow rate was controlled by mass flow controller; each 150 mL/min was used.

Iron(III)phthalocyanine (FePc) was placed in the part of small furnace. At 450°C, 0.1 g of FePc was used for reaction. Hydrogen was introduced from the side of small furnace, and an adapter was placed at the other side to take carbon nanotubes out of the reactor. A paper filter was filled at the gas vent of the adapter. Main furnace was heated up under argon, hydrogen was introduced into quartz tube, small furnace was heated to sublime FePc, then bubbler was activated to the reaction. Main furnace was heated at 800, 1000, 1100, and 1200°C.

SEM (Hitachi S-5000) and TEM (JEOL JEM-2000FX) were used to observe shape and structure of produced carbon nanotube.

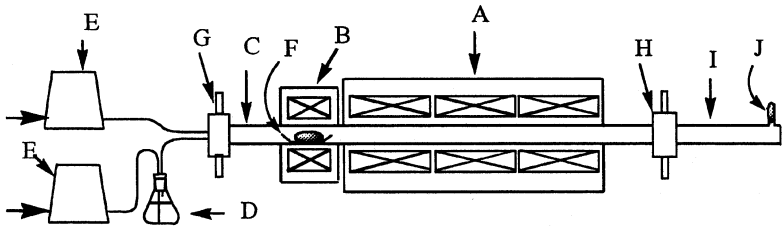

FIGURE 1 Reaction apparatus: A. An electric furnace made by Ta; B. A small Ta electric furnace of 25 cm; C. quartz tube; D. bubbler; E. mass flow controller; F. iron(III)phthalocyanine; G,H. water-cooled flange; I. Adapter; j. paper filter.

RESULTS AND DISCUSSION

Thickness of carbon nanotube was 60 nm to 100 nm at 1200°C.

Produced carbon fiber is shown in Figure 2 at 1100°C. Carbon nanotubes were agglomerated like a ball of wool. The diameter of carbon nanotube is 10 nm to 20 nm, which is equivalent to fast flow synthesis by Ohshima [3]. Main product deposited mainly to paper filter at vent. The reaction was forced to discontinue by clogged deposition.

A resulted carbon nanotube is closely clogged at 1000°C. FePc was passed through quartz tube without any reaction at 800°C.

A longitudinal section of carbon nanotube was observed by TEM. The appearance of observation was shown in Figure 3. nanotubes were obtained. Obtained multi-walled carbon nanotubes were hollow body and even-diameter. The wall consists 5 to 10 layers of solid graphite. Diameter of carbon nanotube is depending on diameter of catalytic particle of 10 to

FIGURE 2 A picture of carbon nanotube produced by synthesis of 1100°C. Thin carbon nanotube is agglomerated like a ball of wool. This nanotube is flexible.

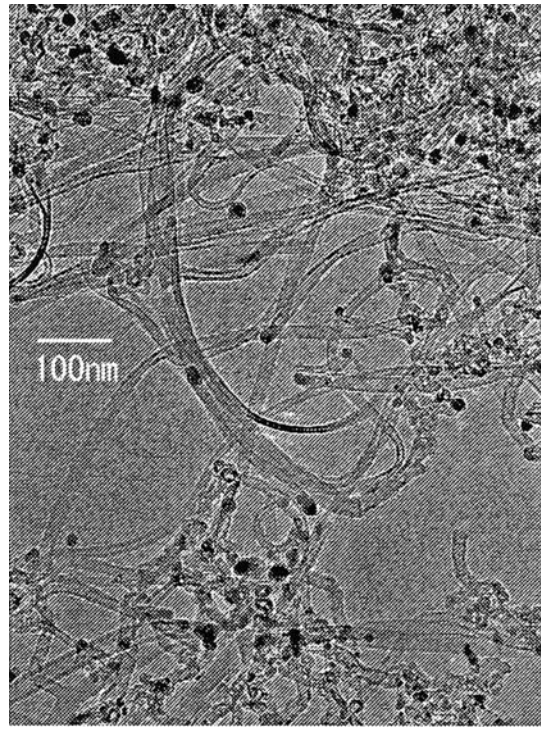

FIGURE 3 A TEM picture of carbon nanotube produced by synthesis of 1100°C. Thinner wall and well identified carbon nanotube was produced in this condition.

20 nm. This carbon nanotube is thick in diameter, however well identified. This carbon nanotube has fare quality by these results.

The carbon nanotube synthesized at this time is expected to act good adsorbent for larger molecules, large adsorption volume and fast adsorption owing to large aperture.

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