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LETTERS
TO THE EDITOR

Synthesis of Carbon Nanotubes from a Cobalt-containing Aerosilogel

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At present carbon nanotubes are one of the most demanded products of carbon nanotechnologies [1–3]. The principal industrial technologies for manufacture of carbon nanotubes are arc discharge, laser ablation, and chemical vapor deposition (CVD) onto the surface of catalysis. The fluidized-bed CVD is used to success for large-scale manufacture of carbon nanotubes [2]. However, thin carbon nanotubes with a narrow size distribution are fairly difficult to synthesize by this technology. Apparently, this problem can be solved not by optimization of synthesis conditions, but also by selection of catalysts with appropriate porous and active-center structures.

In the present work we developed a catalyst for the manufacture of thin multiwall carbon nanotubes by the fluidized-bed CVD technology.

As the carrier for the catalyst we used a porous silica, specifically aerosol gel prepared by the sol−gel method by the procedure described in [4]. It should be noted that silicas are more and more widely used in the synthesis of porous carbon materials, including carbon nanotubes [2, 5]. The specific surface of the aerosilogel as measured by low-temperature adsorption of nitrogen by the BET procedure was 130 m^2/g. The pore size as measured by mercury porosimetry was 25 nm. The pore size distribution was narrow and has a well-defined maximum. Cobalt was deposited on the surface by the ion-exchange procedure described in [6, 7]. The catalyst was synthesized by the hydroxylation of the aerosol gel surface by treatment with dilute HCl, followed by fixation of ammonium cobalt complexes by the ion-exchange procedure involving treatment of the hydroxylated aerosol gel surface with a 0.2 M solution of $CoCl_2$ in an ammonium buffer at pH 8.1 for 20 min. The subsequent hydrogen reduction allowed generation on the aerosol gel surface of metal nanostructures for use as catalysts in the synthesis of thin uniformly sized carbon nanotubes. The cobalt content, according to the atomic absorption analysis, was 0.25 mmol/g.

Carbon nanotubes were synthesized in a quartz reactor equipped with a porous filter. The reactor was placed in a vertical oven with programmed temperature. The synthesis was performed in the fluidized-bed mode at 600–800°C. High-purity hydrogen was used as the reducer and carrier gas, and ethanol was used as the source of carbon. The synthesis procedure involved reduction of the catalyst with hydrogen for 30 min and treatment of the catalyst with ethanol vapors for 30 min. Removal of the catalyst was performed by treatment with HF and HCl. Washing with distilled water gave carbon nanotubes whose electron micrographs are given in Figs. 1 and 2.

The porous structure of the aerosilogel was determined on a PA-3M1 porosimeter. The cobalt content was determined on a MGA-915M atomic absorption spectrometer. The specific surface was measured on a Micromeritics ASAP 2020 MP analyzer. The morphology of the synthesized carbon nanotubes was determined using a Jeol JEM-100C transmission

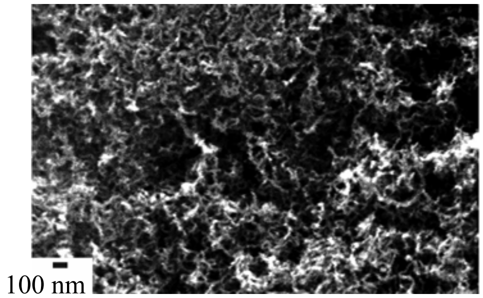

Fig. 1. SEM image of carbon nanotubes.

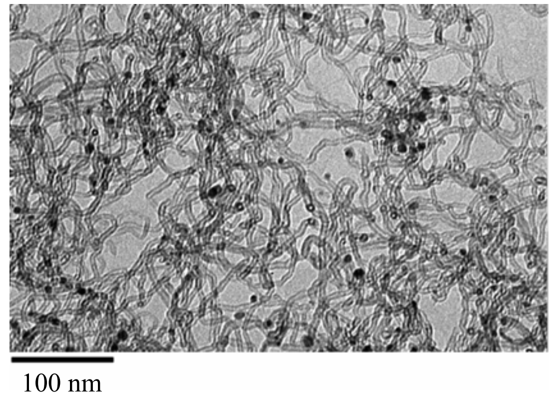

Fig. 2. TEM image of carbon nanotubes.

electron microscope and a Carl Zeiss EVO-40 scanning electron microscope. Elemental analysis was performed on a Shimadzu EDX-720/800HS X-ray fluorescent spectrometer.

Analyzing the images of ultrasonicated carbon nanotubes we can conclude that the use of the cobal-containing aerosilogel makes it possible to manufacture thin carbon nanotubes with the outer and inner diameters of 5–8 and 3–6 nm, respectively. According to the X-ray fluorescent analysis, the carbon product contained 98.9% of carbon, 0.58% of cobalt, and 0.52% of silicon. The specific surface of the carbon nanotubes was 276 m^2/g.

The synthesized carbon nanotubes can present interest as sorbents, chromatographic materials, and fillers for diverse functional polymers, including solid polymer electrolytes for fuel cells.

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