

ELECTROCHEMICAL LI-ION INTERCALATION INTO MULTI-WALL CARBON NANOTUBES

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INTRODUCTION

Since their discovery in the early nineties carbon nanotubes (CNTs) have received broad attention because of their unique physical and electrical characteristics. A number of methods have been used for the synthesis of CNT including arc discharge,^{1,2} laser ablation,^{3,4} and chemical vapor deposition.⁵⁻⁶ Interest in our laboratories have focused on the electrochemical Li⁺ intercalation into various carbonaceous materials as anode materials for rechargeable Li⁺ battery application. This work examines the electrochemical properties of CNT arrays grown directly on an electronically conducting substrate.

EXPERIMENTAL

Multi-walled carbon nanotubes were synthesized on stainless steel (304) 400x400 mesh supporting a thin layer of cobalt as catalyst. The substrates were placed in alumina boats within a linch O.D. alumina process tube. With heating provided by a high temperature tube furnace, the mesh was brought to 800°C under a H₂/He flow. Upon reaching this temperature, a gas mixture consisting of 10 sccm C₂H₂, 25 sccm H₂, 500 sccm He flowed through a bubbler containing benzene and subsequently through the process tube. The thermal CVD synthesis was maintained for a period of 25 min, thereafter the mesh was cooled under a reducing flow, again consisting of H₂ and He. The relative yield as characterized by surface coverage and overall morphology were characterized by SEM, using a Hitachi S4700 instrument with cold-field emission gun. The spatial scale is indicated by the ruler bar, whose full length corresponds to the indicated spatial length.

All electrochemical measurements were performed in 1M LiClO₄ in EC/DEC (1:1 v/v) solution at a scan rate of 0.1 mV/s in a high quality glove box at room temperature using the same chemicals specified elsewhere.⁷ Potential control was achieved using a Pine potentiostat and the electrochemical data collected in a PC via a digital current to voltage converter.

RESULTS AND DISCUSSION

Fig. 1 shows SEM images of CNT formed on Co coated stainless steel mesh surface produced by the method described in the Experimental Section. Typical High-Resolution Transmission Electron Microscopy images of CNT obtained by the flame method, which share common features with those commonly observed with produced with the oven are given in Fig. 2. Fig. 3 shows a cyclic voltammetry curve of CNT(oven) on Co deposited stainless steel mesh substrate in 1M LiClO₄ in EC/DEC (1:1 v/v) solution recorded at a scan rate of 0.1 mV/s. As shown by these data, this specimen showed no evidence for staging as is commonly observed for graphite or carbon fibers; instead, lithium extraction occurred over a wide potential window from 0 to 2.4 V, in agreement with the work reported by Claye et al. for CNT grown by laser ablation in the form of buckypaper.⁸ The ability of the method described in this

work to grow CNT arrays directly on a current collector may prove advantageous for electrochemistry including both energy storage and analytical applications.

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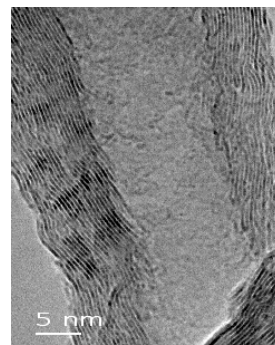


Fig. 1. SEM image of multi-wall carbon nanotubes synthesized by catalytic decomposition of C₂H₂.

Fig. 2. HRTEM image of multi-wall carbon nanotubes.

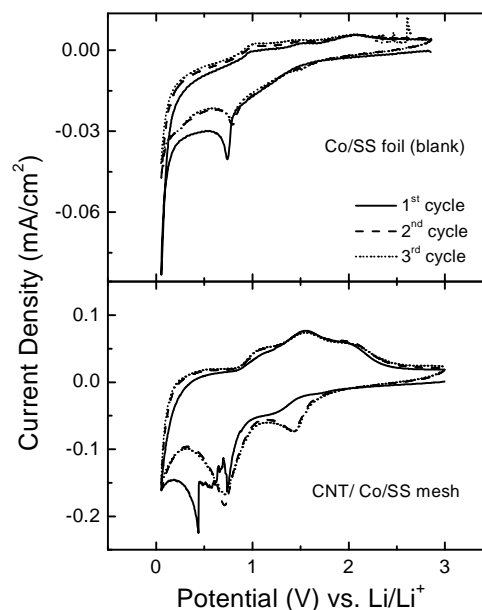


Fig. 3. CV of CNTs synthesized on Co/SS mesh (lower panel) and of Co/SS foil (upper panel) in 1M LiClO₄ in EC/DEC (1:1 v/v) at a scan rate of 0.1 mV/s