SYNTHESIS OF CARBON NANOTUBES ON METALLIC SUBSTRATES BY PECVD AND THERMAL CVD

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INTRODUCTION

Electric double layer capacitors (EDLCs) are attractive energy storage devices particularly for applications that demand high power and long cycle life. The key factors that determine the performance of EDLC are properties of electrodes. Carbon nanotubes can be used for electrodes of EDLC, as they do not contain micro-pores and all surfaces can be available to the access of ions. Synthesis of carbon nanotubes directly on conducting substrates, which can act as current collectors, might greatly simplify preparation of carbon electrodes of EDLC. Carbon electrodes of EDLC could be made from carbon nanotubes grown by thermal chemical vapor deposition (CVD) on conducting substrates pre-coated with metallic catalyst layer (1,2). Carbon nanotubes can also be synthesized directly on metallic substrate by plasma enhanced CVD (PECVD) without pre-coated metallic catalyst layer (e.g. 3).

EXPERIMENTAL

Stainless steel plates (Type 304) were used as substrates for the synthesis of carbon nanotubes. Substrates were mechanically polished, and then etched with HF solution. After treatment with hydrogen plasma synthesis of carbon nanotubes was carried out by rf (13.56 MHz) PECVD with acetylene and hydrogen gases. We could continue growth of carbon nanotubes without plasma, i.e., thermal CVD using acetylene and ammonia gases, after initial growth of carbon nanotubes by PECVD. Deposited carbon nanotubes were examined with SEM and Raman spectroscopy.

RESULTS & DISCUSSION

Etching with HF solution made substrate surface rough baring grains of stainless steel. Fe was etched more than Ni and Cr. Subsequent treatment with hydrogen plasma made grains round in shape and larger in size. Carbon nanotubes synthesized in this study were curly in shape (Fig. 1). One of the reasons for the curly growth of carbon nanotubes might be roughening of substrate surface by etching with HF solution, which can induce incoherent growth direction. Catalyst tip could be observed at the end of some carbon nanotubes (Fig. 1), suggesting that growth of some carbon nanotubes, at least, follow tip growth model. Raman spectroscopy indicated deposition of multi-walled carbon nanotubes with many defects (Fig. 2). Growth rate of carbon nanotubes changed with growth time, and was affected by composition of feed gas. Growth rate of carbon nanotubes by PECVD was relatively high for the ratio of acetylene to hydrogen feed rate of 1:3, but growth stopped in a few minutes. For the ratio of 1:10 initial growth rate was lower, but termination of growth occurred at much later time. For the ratio of 1:30 initial growth rate was even lower and growth of carbon nanotubes continued 30 minutes after the start of the growth. Carbon nanotubes grown further by thermal CVD after initial growth by PECVD were also curly in shape. Thickening of tube diameter was not observed, so weight increase was assumed to be the result of nanotubes lengthening. However, we found that thermal CVD was more effective than PECVD in growth of carbon nanotubes after initial growth by PECVD. Working EDLC could be fabricated from carbon nanotubes synthesized by PECVD and thermal CVD without any further treatment (Fig. 3).

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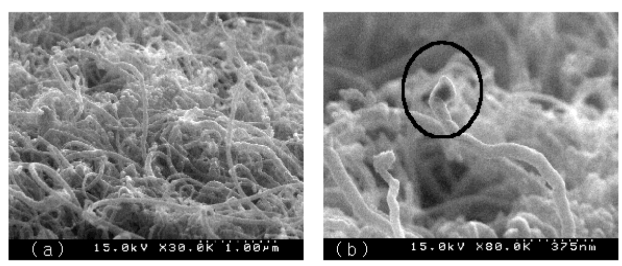

Figure 1. SEM micrograph of carbon nanotubes

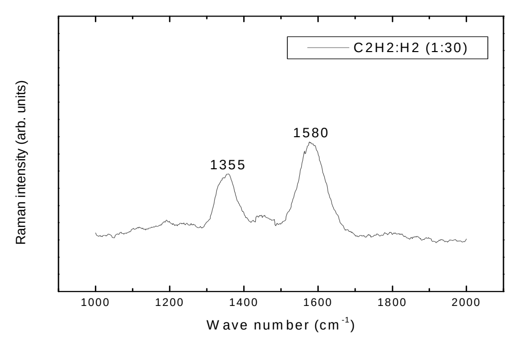

Figure 2. Raman spectroscopy of carbon nanotubes

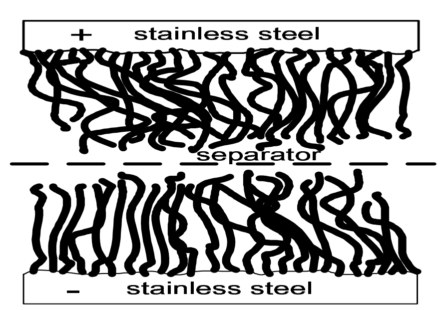

Figure 3. Schematic of EDLC assembly with carbon nanotubes synthesized on stainless steel substrates.