

# ELECTROCHEMICAL PROPERTIES OF MULTIWALL CARBON NANOTUBES

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## 1. INTRODUCTION

Many forms of carbon have been tested as anode in lithium ion battery. Multiwall carbon nanotubes (MWNTs), grown by catalytic and electric arc decomposition of hydrocarbons show large loss of irreversible capacity after first charge [1,2]. Catalytically grown MWNTs show a strong capacitive behaviour in a certain potential range [1]. However, single wall nanotubes (SWNTs), grown by laser ablation, show very high reversible capacity [3]. But the reason behind such high reversible capacity is not fully understood. Studies of Martin et.al, revealed that highly ordered carbon nanotubules show very strong reversible intercalation/deintercalation of  $\text{Li}^+$  and may be suitable as anode in lithium ion battery [4].

In the present investigation, we report on the electrochemical behaviour of MWNTs, synthesized by chemical vapour deposition (CVD) using nanopore alumina template, to elucidate its suitability as anode in Li battery by cyclic voltammetry (CV).

## 2. EXPERIMENTAL

MWNTs were synthesized by CVD of propylene gas using alumina nanopore template at  $800^\circ\text{C}$  using  $\text{N}_2$  as carrier gas. CV was performed using glass cell with three electrode configuration. The working electrode was MWNTs (embedded in  $\text{Al}_2\text{O}_3$ ) ohmically connected to Cu wire by conducting Ag paste. The Ag contact point was insulated by teflon paint which was inert to both the electrolyte and the solvent. Lithium foil connected to Cu wire was used as counter and reference electrode. 1M  $\text{LiClO}_4$  solution in 1:1 volume ratio of EC (ethylene carbonate) and DEC (diethyl carbonate) mixed solvent was used as electrolyte. Ag contact to the WE and ensemble of electrodes in the glass cell was performed in Ar atmosphere using dry box. WE was dried under vacuum at  $120^\circ\text{C}$  for overnight. A potentiostat (model HA301, Hokuto denko Ltd.) with a function generator (HB 104, Hokuto denko Ltd.) was used for carrying out CV experiments. An X and Y recorder

(F-35C, Riken Denshi Co. Ltd.) was used. All potentials are cited with respect to Li/Li<sup>+</sup>.

### 3. RESULTS AND DISCUSSIONS

Fig.1 shows the CV of pristine MWNTs. The open circuit voltage is found to be 2.98 V which indicate that the surface of the nanotubes is almost free from oxygenated species [1].

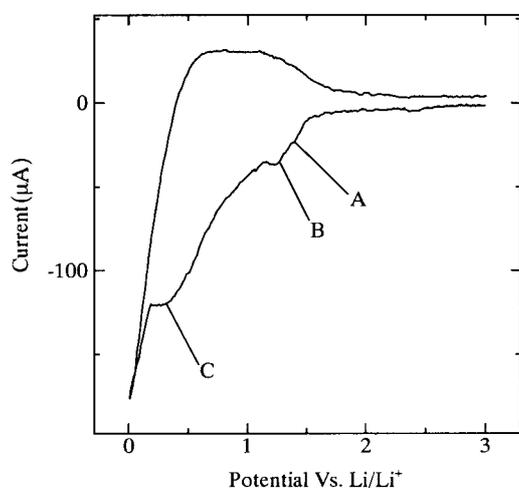


Fig.1. Cyclicvoltammogram of pristine MWNT (1<sup>st</sup> cycle) at a sweep rate of 0.1mVs<sup>-1</sup>.

Four major current peaks are observed in the first discharge scan. Peak A (at 1.4 V) and B (at 1.26 V) can be attributed to the reduction of electrolyte and probably to the formation of solid electrolyte interphase (SEI) [5]. Current peak C may be due to decomposition of solvent to gaseous ethylene and lithium insertion into available nanopores [5]. The discharge scan is completed with a sharp current peak at 0 V due to Li<sup>+</sup> ion intercalation

to MWNTs. On the reverse sweep (first charging) a current plateau, starting at 0.4 V and extended to around 1.5 V is noticed. This plateau may be attributed to deintercalation of Li<sup>+</sup> from various intercalated sites of MWNTs, occurring at different re-oxidation potentials. Moreover, high overvoltage for deintercalation step indicates presence of some kind of attractive force which is hindering it. Effect of heat treatment of MWNTs at 1000°C on its electrochemical property is shown in Fig.2. Current peaks A and B have merged and become much sharp. In the charging scan the current plateau is still observable but the shape is slightly different. It indicates predominance of few intercalated sites than the rests.

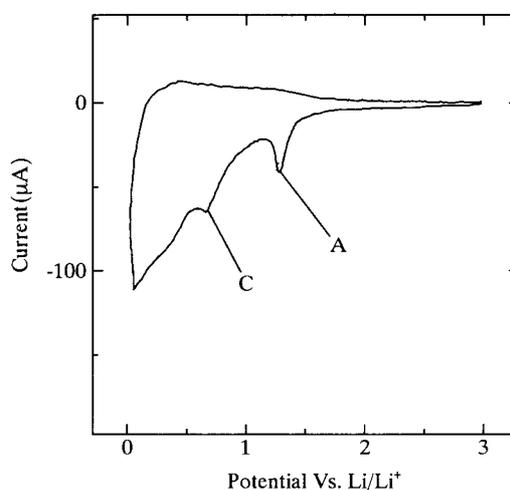


Fig.2. CV of heat-treated (at 1000°C) MWNT (1<sup>st</sup> cycle) at a sweep rate of 0.1 mVs<sup>-1</sup>.

Other successive scans (not shown) further confirms this fact. Unlike

other recently reported results, MWNTs under our investigation don't exhibit any electrochemical capacitive behaviour.

#### 4. CONCLUSIONS

MWNTs synthesized by CVD in the nanopore alumina template show intercalation and deintercalation clearly but the mechanism is seems to be complicated one. The intercalation sites of MWNTs are different with different energetics. The absence of any capacitive behaviour demands further studies of these nanotubes which may be suitable as anode for Li ion secondary batteries.

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