

## Electrochemical characterisation of carbon nanotubes

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### Introduction

Since their discovery in 1991 by Iijima [1], carbon nanotubes (CNTs) have been devised for a number of applications such as hydrogen storage, gas sensors, electrodes for supercapacitors and vacuum microelectronic devices. Among the other properties, it is important to investigate the electrochemical properties of CNTs. In this paper we will present results of electrochemical experiments carried out on carbon nanotubes both obtained from commercial suppliers and produced in laboratory.

### Experimentals

Carbon nanotubes were characterised by cyclic voltammetry and galvanostatic measurements in acidic solutions of  $\text{H}_2\text{SO}_4$  (EG&G 273A, Princeton Applied). Alkaline and neutral solutions were used as well. The counter electrode was a platinum mesh, and SCE as reference electrode was used. Cyclic voltammetry scan rates were in the range  $1 - 50 \text{ mV s}^{-1}$  and constant current ( $10 \text{ mA g}^{-1}$ ) charge-discharge tests were carried out. The cell used is schematically represented in figure 1.

Carbon nanotubes were tested in form of pellets obtained by pressing the powders, after performing a purification step with strong acids. In some cases, despite the purification process of CNTs, residual Fe catalyst particles trapped inside the structure of the nanotubes were not completely removed. The amount of residual Fe catalyst particles contained in some batches of CNTs was exploited to keep the carbon nanotubes in form of powder attached to a gold foil current collector by the action of strong permanent magnet. Both the current lead and the permanent magnet were coated with NiP and finally with gold (Fig. 2). Gold was used for its inertness, thus preventing any undesired interaction between the solution and the current collector.

### Results

A typical cyclic voltammogram obtained at  $5 \text{ mV s}^{-1}$  of scan rate in  $\text{H}_2\text{SO}_4$  (0.3 M) is displayed in Figure 3. Broad reduction and oxidation peaks centred at around  $0.3 \text{ V vs SCE}$  can be observed. The shape of the voltammogram is similar to that obtained by Barisci *et al* in similar conditions [2]. The oxidation and reduction peaks can be related to the release and uptake of hydrogen on the oxygen-containing functional groups of the nanotubes.

The cut-off potential of the galvanostatic experiments was fixed at  $1 \text{ V vs SCE}$ . Typical charge-discharge curves are shown in figure 4. It is possible to observe a change of slope in the discharge curve at  $0.3\text{-}0.4 \text{ V vs SCE}$ . This feature may be related to the peak observed in the cyclic voltammogram.

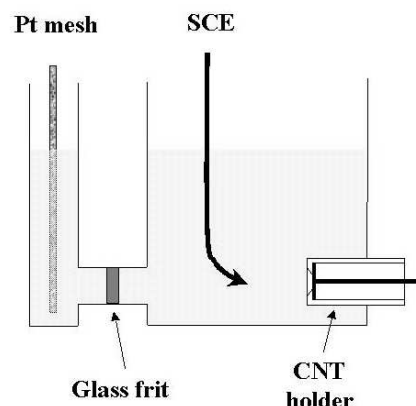


Fig. 1. Electrolysis cell.

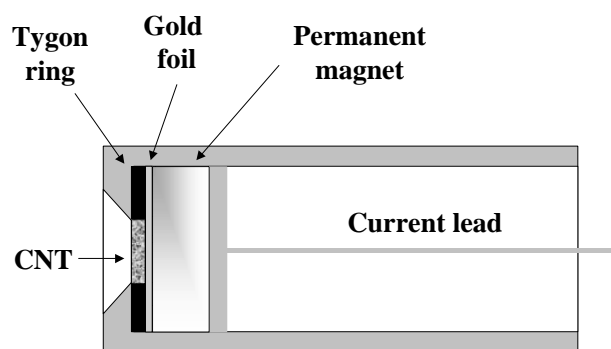


Fig. 2. CNT holder.

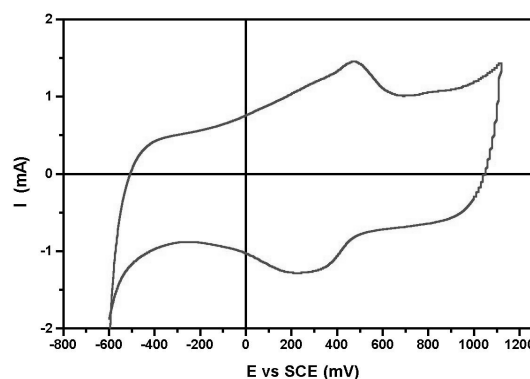


Fig. 3. Cyclic voltammogram of a CNT sample at  $5 \text{ mV/s}$  scan rate.

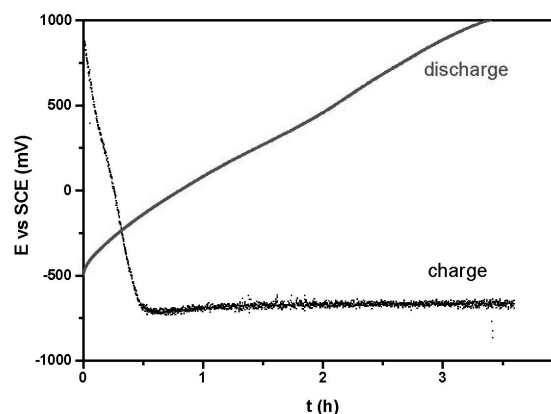


Fig. 4. Charge-discharge curves of a CNTs sample.

### References

- [1] P. Iijima, *Nature* 354 (1991) 56-58.
- [2] J.N. Barisci, G.G. Wallace, R.H. Baughman; *Journal of Electroanalytical Chemistry* 488 (2000) 92-98.

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