Filling carbon nanotube membranes with Pd and TiO_2

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The design of novel hybrid nanostructures, with specific functionalities and well defined dimensions are among the desired properties of many emerging applications. Learning how to grow nanostructures, in specific locations with certain morphology and functionality, remains, in many areas, a challenge for the continuous progress of technology. With the aim of contribute in this subject we will present the synthesis and characterization of palladium and titanium dioxide nanostructures grown selectively inside carbon nanotubes.

Carbon Nanotubes were synthesized by decomposition of acetylene inside the pores of anodized aluminum oxide (AAO). Two types of membranes were used: one prepared by the anodization of an Aluminum foil to obtain a self-supported material (~ 50μm thick), the other one was prepared by the anodization of a thin Aluminum film deposited on silicon chip by e-beam evaporation (≤ 1μm thick).

The grow of CNTs was performed at 650 ºC with a mixture of Argon/acetylene (200/25 sccm) by periods of 10-30 min. Figure 1(a) show a SEM image of the obtained CNTs after a partial AAO removal. Figure 1(d) shows a TEM image of the CNTs obtained using AAO-Si membrane after total removal of AAO.

Since the tubes outside walls are initially completely covered by the template, we can very easily access the inside the tubes by molecules or metals precursors in liquid dissolutions, while the outside wall remains free of any molecules or particles. As an example of the potential uses of these membranes as nanoreactors, we have explored the possibility to form palladium and TiO_2 nanostructures inside the carbon nanotubes.

To prepare palladium composites we have used $PdCl_2$ and $Pd(NO_3)_2$ dissolutions to impregnate the CNT-AAO membranes by dip-coating or by drop-casting. After impregnation the membranes were calcinated (350 ºC) in an O_2/Ar mixture and reduced (450 ºC) in H_2/Ar atmosphere. The AAOs were finally removed with a NaOH solution, leaving behind nanotubes filled with nanoparticles (see figure 1 b and c). Preliminary results of the electrical characterization of these Pd@CNT nanohybrid films deposited over interdigitated microelectrodes indicate that these materials could be used as gas sensors since their resistivity change considerably with different gas mixtures.

On the other hand, TiO_2-CNT composites were prepared by Chemical Vapour Deposition (CVD) of a conventional titanium precursor [1], Titanium Tetraisopropoxide (TTIP), over CNT-AAO-Si membranes. The TTIP was introduced to the tube furnace by bubbling Argon through a vessel with the titanium precursor previously thermalized at 100 ºC, and then decomposed at 400 or 500 ºC. The TEM results show that TiO_2 effectively cover the inner cavity carbon nanotubes (see figure 1 e and f).

The results show that the use of AAO membranes is very useful to prepare nanostructures selectively inside carbon nanotubes at difference of other methods used by our group [2, 3].

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References

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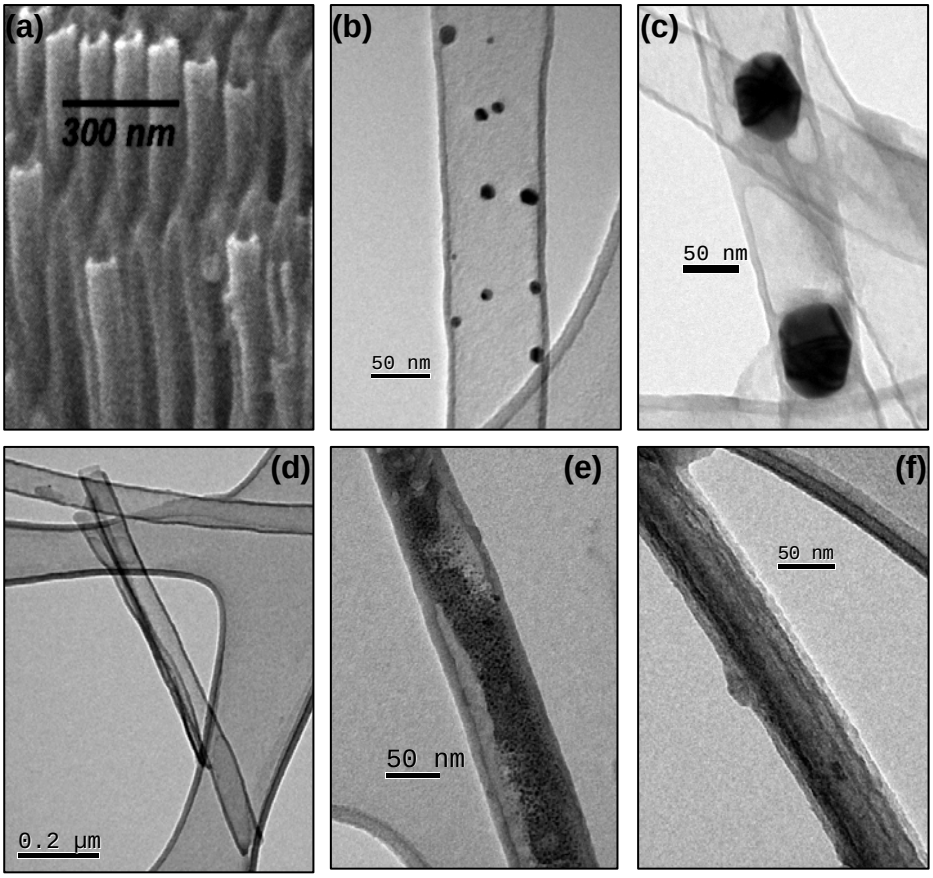

Figure 1. Electron microscopy Images of (a) CNTs with AAO partially dissolved, (b) Pd@CNTs prepared by dip-coating in $PdCl_2$ precursor, (c) Pd@CNTs prepared by drop-casting with $PdCl_2$ precursor, (d) CNTs prepared in AAO-Si membrane, (e, f) TiO_2-CNTs nanohybrids.