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2D-TEM investigations of CNTs synthesized within vertical-PAA templates for devices applications

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Since their discovery in 1991 carbon nanotubes (CNTs) become one of the most emblematic type of nanostructures due to their unique electrical, mechanical, thermal and optical properties. Despite these promising and astonishing properties, the critical subject of their manipulation remains a primordial challenge. Very expensive and time consuming techniques, like e-beam lithography, have been developed in order to precisely control their growth, or to obtain a nano-object from the etching of a bulk material. Such techniques are suitable as proofs of concept; however the object-by-object manipulation approach is unrealistic because they are not compatible with a massive integration. On the other hand the miniaturization of the fabricated devices is reaching a bottleneck since the scaling-down for enhancing their performance is approaching many physical limitations. The efforts concentrated to achieve device fabrication by continuous miniaturization are nowadays struggling with performance decline in the electrical conductivity transports values and a significantly loss of reliability. Self-organized templates such as porous anodic alumina (PAA) templates provide several advantages for controlling the nanostructures growth.[1,2] Its well-ordered structure and the confinement imposed by the nanopores the PAA template offer a promising approach for cost-effective, stable and efficient fabrication of carbon nanotubes based devices.[3]

Here we present a complete 2D analysis based on advanced electron microscopy techniques devoted to the full characterization of both the PAA structure and the as-grown CNTs using a dHF-CVD (double-Hot Filament assisted CVD) synthesis method. More exactly, we combine the FIB (Focused Ion Beam) preparation technique with advanced TEM characterization techniques such as STEM-EDX and EELS spectroscopy for the assessment of an accurate correlation between the synthesis parameters and the morphological, structural and chemical characteristics of both the PAA structure and the as-grown CNTs.

In a first step, the TEM analysis of different PAA cross-sections prepared using the FIB technique, allowed us accessing precise characteristics such as the pore length (800nm) and their diameter (30nm) as well as the inter-pore distance (30nm) (see figure1). A more detailed analysis on the bottom part of the PAA structure helped us evidencing the presence of a branched nanopores structure product of an exponential voltage decrease process, applied in order to thin the oxide barrier layer at the bottom of the pores.

For the CNTs, we first examined the impact of the catalyst pretreatment step performed prior to the CNTs growth step. Secondly, by varying the hot-filaments power applied during the growth, we investigated the impact of the additional gas phase activation conditions over the synthesized carbon nanostructures. The results revealed that the pretreatment conditions determine the catalyst distribution at the bottom pores of the PAA membranes, with a strong impact on the CNTs growth within the PAA templates. Another important finding concerns the amount of defects incorporated into the grown CNTs walls which could be related to the hot-filament power applied during the synthesis.

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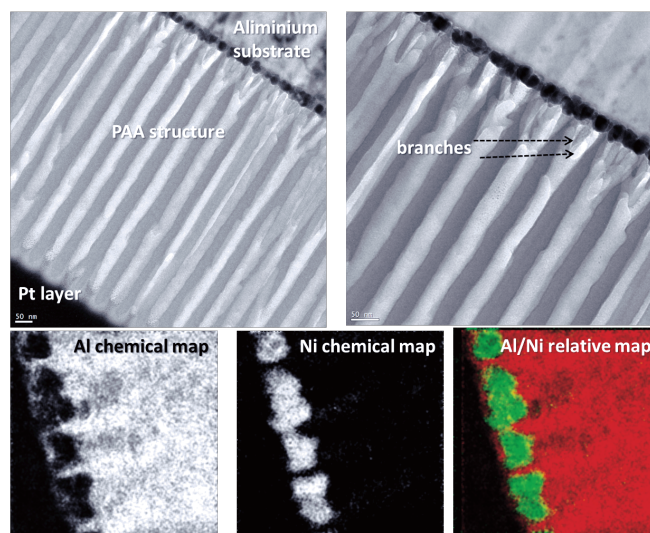


Figure 1:Up-left: TEM image of a PAA cross section illustrating its well-organized structure. Up-right Zoom on the pores bottom part evidencing the presence of branched nanopores structure. Bottom:Chemical analysis of the PAA/Ni structure with Al chemical map (left), Ni chemical map (middle) and Al/Ni relative map (right).

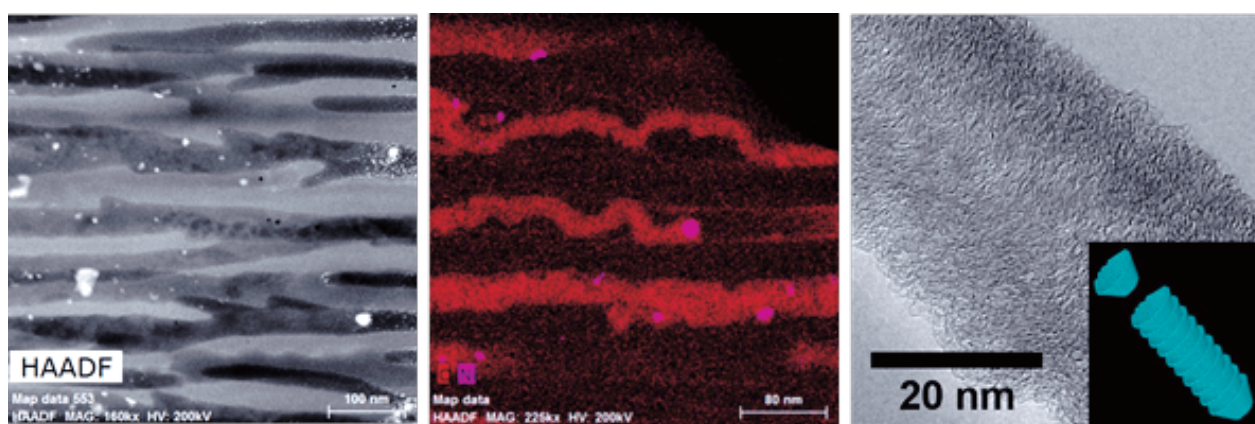


Figure 2: Left: HAADF-STEM image for the chosen area of PAA/Ni where the EDX chemical analysis has been performed; Middle: C/Ni Relative map with the C in red and Ni in pink; Right: HRTEM image of the CNTs outside the PAA structure illustrating its cup-stack structure with in inset a schematic diagram of the cup-stack structure.