Structural and EELS studies on doped carbon nanostructures for Cold-Field Emission Guns

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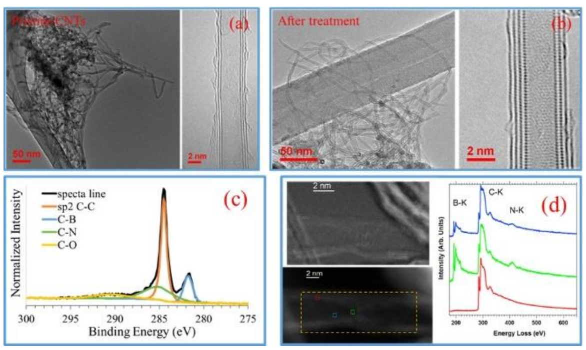

Fig. 1 (a) TEM images of pristine and (b) treated CNTs. (c) XPS spectra of C1s of these nanomaterials. (d) STEM images and selection of EEL spectra recorded on this CNT.

So far, cold field emission (C-FE) is considered as the best way to emit electrons in transmission electron microscope (TEM). This is due to their excelent spatial and temperal coherence. However, the use of conventional tungsten emitter is still inadequate due to its large emission area and the need of flash. Therefore, it is imperative to developpe new cold cathod materials. Carbon nanotubes (CNTs) and nanocones have been considered as very promising candidates for C-FE[1,2]. This research is devoted to the doping of these carbon nanostructures by nitrogen and/or boron in order to ameliorate their emission perfomances.

CNTs were heated in a tubular furnace from 1300°C to 1500°C under various atmospheres. The deep studies of their structure and atomic composition have been developed by high resolution TEM and spatially-resolved electron energy loss spectroscopy (EELS) in scanning (STEM) mode at 80 kV. In addition, X-ray photoelectron spectroscopy (XPS) has also been carried out. Through these characterizations, we have concluded that after the thermal treatment: (1) no significant structural modifications are observed (Fig.1 (a), (b)), and (2) the formation of C-B and C-N bonds is produced (Fig.1 (c)). Furthermore, from the EELS studies we have demonstrated, at the local scale, the formation of boron nitride nanodomains and of hybrid nanotubes (Fig.1 (d)). The influence of the introduced hetero-elements in the carbon nanostructures is clarified in the DFT calculations.

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1. Gröning, O. et al. Field emission properties of carbon nanotubes. Journal of Vacuum Science & Technology B 18, 665–678 (2000).
2. Houdellier, F. et al. *Development of TEM and SEM high brightness electron guns using cold-field emission from a carbon nanotip*. Ultramicroscopy 151, 107–115, 2015.